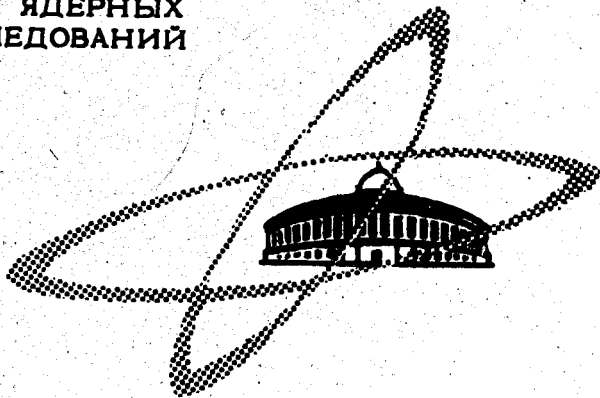


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ОБЪЕДИНЕННЫЙ  
ИНСТИТУТ  
ЯДЕРНЫХ  
ИССЛЕДОВАНИЙ

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EXPERIMENTS ON CHEMISTRY  
OF ELEMENT 104-KURCHATOVIIUM.  
V. ADSORPTION OF KURCHATOVIIUM  
CHLORIDE FROM THE GAS STREAM  
ON SURFACES OF GLASS  
AND POTASSIUM CHLORIDE

ЛАБОРАТОРИЯ ЯДЕРНЫХ ИССЛЕДОВАНИЙ

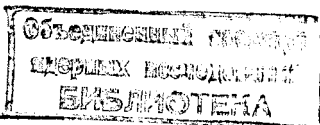
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Submitted to Inorg. Nucl. Chem.



\* The experimental results were reported by I.Zvara at the 155th ACS National Meeting, San Francisco, April 1968.

## Introduction

Earlier<sup>/1,2/</sup> our group reported the chemical identification of the element number 104-kurchatovium. This was performed by comparing the volatility of kurchatovium chloride with that of chlorides of group III and group IV elements. There were described the method<sup>/3/</sup> and equipment<sup>/4/</sup> used in this study.

Element 104 was synthesized on an atom-by-atom basis by irradiation of a plutonium-242 target with accelerated neon-22 ions<sup>/5/</sup>. The volatility of kurchatovium chloride was estimated from measuring adsorption of its molecules on solid surfaces. The characteristic features of the experiments were as follows (cf. also Fig.1): Atoms recoiling from the target due to the momentum imparted by impinging ions were stopped in flowing inert gas and then chlorinated downstream. The chloride molecules passed through a chromatographic column with radiation detectors at its outlet. A total of 14 kurchatovium atoms were observed by detecting fragments from spontaneous fission of their nuclei. Retention time data served for estimating the heat of adsorption values for the chlorides to be

compared. It was shown, that according to the chloride volatility, the new element is the homologue of group IV elements (hafnium and zirconium) and sharply differs from group III elements (californium, fermium). Recently<sup>/6,7/</sup>, it was demonstrated by direct experiments, that element 102-joliotium and element 103 behave, at the same conditions, as elements of the third group and thus they are "ekaytterbium", and "ekalutecium", respectively.

In the present work we continued the study of the compound of kurchatovium with chlorine to get more accurate data and to search for additional evidence of tetravalency of element 104. We measured the adsorption of kurchatovium chloride at 300<sup>o</sup>-350<sup>o</sup> on glass and on KCl crystals from a gaseous mixture of chlorine containing species. For comparison, we followed in the same experiments the behaviour of chlorides of fermium, californium, curium, americium, plutonium, scandium, and sodium. For measuring the adsorption of chlorides of a number of other elements under comparable physico-chemical conditions, we utilized a different apparatus working out of the cyclotron beam with use of commercial tracers.

## EXPERIMENTAL

### Apparatus

The gas-solid chromatography apparatus on the extracted beam of the JINR cyclotron U-300 is diagrammed in Fig.1 (The earlier work<sup>/1,2/</sup> was performed with a different equipment<sup>/4/</sup> on the internal beam of this accelerator). The target consisted of a plutonium or americium oxide layer 0.8 mg/cm<sup>2</sup> in thickness, supported by a 9  $\mu$  aluminium foil. Nitrogen was passed through the volume behind the target with a flow rate 5 l at N.T.P./min). At working pressure of

1.15 atm. and temperature of  $300^{\circ}$ - $350^{\circ}$ , the estimated range of recoiling kurchatovium atoms should be about 10 mm. The volume was 20 mm deep with a conical outlet part. Through an orifice the atoms, swept by the gas, entered the chlorination chamber. This was a molybdenum glass 60 mm x 8 mm i.d. tube. The chamber was joined with a 650x2 mm i.d. capillary tubing, welded into a wider outside glass jacket. This served for introducing into the chlorination chamber a stream of nitrogen (0.3 l/min) carrying vapors of "chloride carriers"<sup>[3]</sup> - thionylchloride, niobium pentachloride and zirconium tetrachloride. Partial vapor pressure of these chlorides resulting from mixing the streams was 60, 0.03 and 0.005 mm of Hg respectively. For maintaining the steady concentration, the chlorides were introduced continuously into the apparatus through a dosing device as a liquid solution of  $NbCl_5$  and  $ZrCl_4$  salts in  $SOCl_2$ . This solution was evaporated in the jacket. The apparatus was constructed so as to allow also, if desirable, the gas introduced into the chlorination chamber to be saturated with  $NbCl_5$  and  $ZrCl_4$  vapors by passing it through a bed of the solid substances<sup>[2]</sup> (not shown in Fig.1). The vertical part (see Fig.1) of the column consisted of a 100 mm x 5 mm i.d. section with a somewhat increased inner surface and of a 550 mm x 2 mm i.d. tubing. Thus the apparent surface of the whole column was about  $120\text{ cm}^2$ , the free volume  $10\text{ cm}^3$  and the carrier gas transit time 0.065 sec. The reason for placing the second half of the column under the  $90^{\circ}$  angle to the first one was the necessity of effective shielding the mica detectors against the neutron flux generated by the ion beam striking the target and the target holder. The detectors were placed in a steel-made chamber, connected with the outlet of the glass column. The inner volume of the chamber was 600 mm in length, 60 mm in width and 6 mm in height. Mica sheets of the  $720\text{ cm}^2$

overall area covered both horizontal surfaces. The pressure in the chamber was close to the atmospheric one, the temperature 280°, the carrier gas transit time 1.5 sec.

### Background of detectors

There were two possible sources of the "background" fission fragment tracks: the induced fission of uranium contained in mica by neutrons and the contamination of detectors by the target material spontaneously fissioning plutonium-242, in the course of the experiment. As the shielding against neutrons there were used 300 mm of iron, 300-400 mm water jacket and wrapping the steel chamber with 0.5 mm cadmium (not shown in Fig.1). The integral neutron flux in the detector chamber was measured in each experiment using an uranium reference specimen in contact with mica. It was found that the neutrons could produce one fission in uranium, contained in mica, per 40 days of experimentation.

The amount of plutonium on the detectors and beyond them in the gas duct was determined through measuring plutonium-238. This was the component of the target material (see below), being the source of its rather high specific alpha-activity ( $10^7$  d/mg sec). After finishing an experiment, all mica sheets were counted with the  $2\pi$  gridded ionization chamber. As low as  $10^{-4}$  fraction of the "dangerous" level of contamination could be detected. According to these measurements, not more, than one fission event in all experiments might be attributed to this source of background.

### Synthesis and identification of radioisotopes

The energy of the extracted ion beam was measured by absorption in aluminium foils to  $\pm 1.5\%$ . It was changed by degradation in aluminium foils placed in front of the target. The beam current which passed through the target was integrated with a Faraday cup device.

The authors of the nuclear physical evidence-based identification of element 104<sup>/5/</sup> assigned the spontaneously fissioning activity (  $T_{1/2} \approx 0.3$  sec) they observed, to mass number 260. They thought the shape and position of the maximum of the experimental excitation curve were consistent with the parameters to be expected in the case of the reaction  $^{242}\text{Pu} ( ^{22}\text{Ne}, 4n ) ^{260}104$ . Later it was made clear, that the maxima of the excitation curves for reactions with the evaporation from compound nuclei of 3, 4 and 5 neutrons should fall in the narrow range (a few MeV) of the excitation energy<sup>/8,9/</sup>. This should result in the presence of more than one isotope of element 104 in the target bombardment products and, hence, it cannot be excluded, that there was some incorrectness in the original identification of the mass number of the 0.3 sec activity.

The isotopic composition of plutonium, used for making the targets, was (atom percentages):  $^{244}\text{Pu}$  0.1,  $^{242}\text{Pu}$  92.6,  $^{241}\text{Pu}$  1.5,  $^{240}\text{Pu}$  3.2,  $^{239}\text{Pu}$  1.2,  $^{238}\text{Pu}$  1.4. The composition of americium was:  $^{243}\text{Am}$  94,  $^{241}\text{Am}$  4.9,  $^{242,244}\text{Cm}$  1.1.

The excitation curve for the formation of the 0.3 sec kurchatovium activity was reported to peak at  $^{22}\text{Ne}$  ion energy of 114 MeV with the half-width of 10 MeV, the maximum cross section value being  $\approx 1 \cdot 10^{-34}$  cm<sup>2</sup>. The maximum yield to be expected for the utilized target ( $1.7 \cdot 10^{18}$  atoms of  $^{242}\text{Pu}/\text{cm}^2$ ) was one atom per  $5 \cdot 10^{15}$

$^{22}\text{Ne}$  ions passed through the target. Among the products of the bombardment of plutonium there are found two other spontaneously fissioning nuclides<sup>/5/</sup>. One of them was identified as  $^{242\text{mf}}\text{Am}$  (  $T_{1/2} = 14$  msec), the atomic and mass number of the other one (  $T_{1/2} = 8$  sec) is not known. This appears to be an isotope of some transuranium element. The interaction of bombarding ions with target nuclei results in formation of a number of long lived  $\alpha$ -active isotopes of transuranium elements, through nucleon

transfer (incomplete fusion) reactions. Due to a minute sputtering of the target material by the ion beam, the target constituents were also introduced into the gas stream.

In the present experiments we followed the behaviour of  $^{252}\text{Fm} + ^{255}\text{Fm}$ ,  $^{246}\text{Cf}$ ,  $^{240,242,244}\text{Cm}$ ,  $^{241,243}\text{Am}$ ,  $^{238}\text{Pu}$ , as well as  $^{44}\text{Sc}$  and  $^{24}\text{Na}$ . (The latter two were formed in nuclear reactions on aluminium<sup>/10/</sup>). For this purpose, after ending an experiment, the apparatus was dismantled and the distribution of the above long lived isotopes was measured along the whole gas duct. Procedures used for chemical separation of radioelements and for identification of isotopes by their alpha decay and gamma ray energies and half-lives, were described in detail in <sup>/10,11/</sup>.

### Materials

Commercial 99.9% nitrogen was further purified from oxygen using active copper and dried over molecular sieves 5A. According to the chromatographic analysis, the content of oxygen was lowered to 0.005%. "Technical pure" grade thionylchloride, "spec pure" niobium pentachloride with 0.035% Ta and "pure" zirconium tetrachloride, sublimed in hydrogen stream and in vacuo, were used.

## RESULTS AND DISCUSSION

A total of twenty separate 40-50 hrs runs were performed. They are grouped in four series and the results are shown in Table I. Its last four columns give: the integral flux of particles having passed through the target,  $I$ ; the calculated number of atoms of the 0.3 sec isotope produced,  $N_0$ ; the number of fission events detected,  $N$ , (the number in the brackets refers to the



events when both fragments were detected on the opposite mica sheets) and the ratio  $\frac{N}{N_0}$ .

In series I-III, the plutonium target was employed. The energy of  $^{22}\text{Ne}$  ions in different runs was within the range of 113 to 118 MeV. It will be seen, that in the experiments of series I and II the ratio  $\frac{N}{N_0}$  is somewhat above 10%. To check that the observed fission events were owing to kurchatovium and cannot be explained by other spontaneously fissioning products (see above) we carried out some control irradiations. We changed the  $^{22}\text{Ne}$  ion energy and made use of different targets (series IV) to increase the yield of various possible spontaneously fissioning nuclides, while suppressing the formation of element 104.

In two experiments the plutonium target was bombarded by the  $^{22}\text{Ne}$  ions with the energy of 129 MeV. Compared with 114 MeV, this should result in the about ten fold increase in the yield of the 0.014 sec and 8 sec activities and in the ten fold increase in the yield of the 0.3 sec isotope  $^{15}\text{I}$ . With the integral flux of  $6 \cdot 10^{17}$  ions, two fissions were detected, i.e. eight times less than for the same flux of the 114 MeV ions, this being consistent with the estimate

$N_0 = 12$ . These data provide evidence, that the spontaneously fissioning nuclides of the elements which form involatile chlorides, are separated to a high degree.

Nevertheless, as the retention time of lanthanide and transuranium element trichlorides is of the order of the duration of an experiment, some fraction of atoms of the long-lived isotopes of these elements passed through the column and could be detected beyond it in the gas duct. From the summarized data of series II experiments this fraction was (with standard error from counting statistics):  $6.6 \pm 1.0\%$  for  $^{252}\text{Fm} + ^{255}\text{Fm}$ ,  $3.4 \pm 0.3\%$  for  $^{246}\text{Cf}$  and  $3.9 \pm 0.1\%$  for  $^{242}, ^{244}\text{Cm}$ . Unlike the present findings, the results

of earlier studies<sup>/7/</sup> did not imply greater transfer of fermium chloride itself through the column as compared with curium and californium chlorides. So the "surplus" fermium atoms might be genetically related to the isotopes of element 104 with mass numbers 259 and/ or 260. This uncertainly originates from the impossibility of resolving the  $^{252}\text{Fm} + ^{255}\text{Fm}$  mixture by radiometry at low counting rates, as these isotopes posses almost equal alpha particle energies and half-lives. At present, the possible modes and chains of decay of  $^{260}104$  and  $^{259}104$  are not sufficiently investigated so the above observation needs further studies.

Next, there were performed some bombardments of plutonium and americium targets with the  $^{18}\text{O}$  ions. In these combinations no nuclei with  $Z=104$  can be formed, while there should be a considerable increase in the yield of many nuclides with  $Z < 104$ <sup>/5/</sup>. Only two fission events were detected at the integral flux of  $8.6 \cdot 10^{17}$  particles. These experiments, as well as the above discussion of the sources of background tracks, provide evidence that the fission events observed in series I and II were almost completely due to spontaneous fission of the element 104 nuclei.

### Half life

In principle, the described detector assembly can be used for estimating the half-life. In the bottom part of Fig.2 there is presented the distribution of fission events along the detector chamber, as observed in the experiments of series II, in which most of fissions were detected. In the top part of this figure it will be seen a "decay curve". This was drawn with the assumption, that the kurchatovium chloride molecules move through the chamber with the average linear velocity of the gas stream, and so, that the change in frequency of the tracks per path length unit presents a true pic-

ture of the decay of the isotope in time. The quantity A in Fig.2 plotted against the time is the full number of the fission events detected beyond a given detector, this corresponding to some time interval. These data imply the half-life of somewhat less than one second. The total number of fission events observed in refs. 1 and 2 and in the present bombardments of plutonium with the  $^{22}\text{Ne}$  ions, was 79. Their distribution plotted at the above assumption gives the half-life of about 0.5 sec. This is close to the value found for a kurchatovium isotope by Flerov et al.<sup>15/</sup>

The validity of the assumption involved in the half-life estimation cannot be, however, considered as firmly established. It is possible, that the molecules of kurchatovium chloride pass through the chamber more slowly than the carrier gas does. If so, then the experimental half-life might be lower than the true one.

#### Retention time and heat of adsorption

In five experiments of the series II (23 fission events observed) there was measured also the distribution along the detectors of the fraction of scandium and plutonium atoms (3% and about 15% respectively), transferred into the chamber in the course of the experiment. These elements form involatile chlorides, which are strongly adsorbed. The distributions (in percentages) are presented in Fig.3 in an analogous manner as the top part of Fig.2. It will be noted, that the distribution of fission tracks is less steep. This, in itself, indicates much weaker adsorption of kurchatovium chloride, as compared with chlorides of scandium and plutonium, and provides an additional evidence, that the observed fission tracks could not be due to the partial transfer through the column of isotopes of involatile-chloride-forming elements.

The retention time,  $t_R$ , of a short-lived isotope in the column can be estimated from the evident relation

$$N = \eta N_0 e^{-\frac{t_R \ln 2}{T_{1/2}}}$$

The coefficient  $\eta$  is introduced to take into account:

- the loss of those kurchatovium atoms, which did not recoil into the volume behind the target ( $\approx 10\%$ ),
- the loss due to the irreversible adsorption of the atoms on surfaces of this volume (found to be 10% for Cm, Cf, Fm and Hf atoms),
- the overall efficiency of the detector assembly ( $\approx 80\%$ ).

Taking 0.5 sec for the half-life, the values of  $t_R$  were calculated to be 1.7 sec and 1.4 sec in series I and series II, respectively.

It should be noted, that the ratio  $\frac{N}{N_0}$  and, hence, also the calculated value of  $t_R$  does not change considerably, when increasing the column temperature from  $300^\circ$  to  $350^\circ$  (see Table I). This is in contrast with the earlier observed sharp increase in  $\frac{N}{N_0}$  when going from  $250^\circ$  to  $300^\circ$ <sup>1,2/</sup>. Apparently, increasing the temperature leads to a weaker modification of the surface because of diminishing its fraction, covered by the adsorbed chloride carriers. Then, above some temperature, the resulting increase in the heat of adsorption of the trace component to be eluted, might compensate the speeding action of the higher temperature.

Another explanation for the constancy of  $t_R$  might consist in that the value accepted for the quantity  $\eta N_0$  was too high. From the above values of  $t_R$ , making use of a molecular-kinetics equation<sup>4/</sup> one obtains for the heat of adsorption of kurchatovium chloride  $Q_a \approx 24$  kcal/mol (at  $300^\circ$ ).

Using the data on the behaviour of other elements followed in these experiments and results obtained with the apparatus for work with radioactive tracers, we calculated the values of heat of adsorption for chlorides of a number of known elements. All these values are related to the surface of glass, modified by contact with vapors of thionylchloride, niobium pentachloride and zirconium tetrachloride. It was found, that there is a linear relationship between the values of heat of adsorption and those of heat of vaporization, as shown in Fig.4. The straight line was drawn on the basis of heat of adsorption data for chlorides of elements Nb , Hf , Hf , In , Th , Na, Sc and Am . There is almost no doubt concerning the chemical state of these elements in the gas stream, which is indicated in the figure. Heat of vaporization values for the corresponding species are available. In the case of Pa , U and Pu one hardly can be so sure and the measured heats of adsorption are placed on the straight line to show the position of these elements in what might be called the "adsorption row".

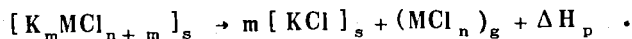
The linear correlation of Fig.4 can be used for estimating the volatility (heat of vaporization) of chlorides of new elements. For kurchatovium chloride, from  $Q_a = 24$  kcal/mol the value of L is found to be about 21 kcal/mol.

In a recent work<sup>[7]</sup> there were determined experimentally some lower limits of the possible values of heat of adsorption for the chlorides of element 102 and element 103. These values are 30 and 33 kcal/mol respectively, implying low volatility of the compounds.

#### Interaction with potassium chloride

As was shown above, the relation of heats of adsorption or of retention times reflects the relation of heats of vaporization.

There is some evidence<sup>/11/</sup> that, similarly, the relative behaviour of various volatile chlorides when passing through a bed of potassium chloride can be correlated with the relation of enthalpies of decomposition ( $\Delta H_p$ ) of the complex compounds, which are formed in the systems under consideration. The reaction in general form is



Consequently one may expect, that changing the glass column for the column of KCl the increase in retention time will reflect the difference  $\Delta H_p - L$ .

According to the available thermochemical data<sup>/14-17/</sup>, this value for  $KLnCl_4$ ,  $K_2HfCl_6$  and  $KNbCl_6$  is approximately 10, 30 and 10 kcal/mol, respectively. Hence, the retention time of the chlorides of group IV elements is expected to increase by a high factor.

This proved to be the case. One experiment of series III was carried out with one third of the column replaced by the capillary made of potassium chloride and at the integral flux of  $3.4 \cdot 10^{17}$  particles no fission tracks were detected. At the same time, the retention time of Am, Cf and Fm isotopes only slightly increased (the transfer through the column diminished to 1%) and niobium pentachloride passed through the column without interaction. Thus, kurchatovium chloride, like the highest chloride of hafnium<sup>/11/</sup> and in contrast with the chlorides of trivalent and pentavalent elements, shows an increased adsorbability on the surface of potassium chloride. This points to  $KuCl_4$  as the most probable state of kurchatovium.

## Conclusion

The results of the present work confirmed, that kurchatovium forms with chlorine a tetrachloride, which is similar in properties to  $\text{Hf Cl}_4$ , and that, consequently, the new element, as the homologue of hafnium and zirconium, is the member of IV B group of Periodic System. This is in agreement with the main conclusion drawn from the first set of experiments of kurchatovium chemistry<sup>[1,2]</sup>.

The body of results of the present and previous works provides the experimental evidence, that 5f -element family ends with element 103 in the same sense as the 4f -element family ends with lutecium, and so, that the seventh period of the Mendeleev system is of the analogous structure as the sixth one.

While  $\text{Zr Cl}_4$  and  $\text{Hf Cl}_4$  differ only very slightly, kurchatovium chloride seems to be somewhat less volatile than hafnium tetrachloride. If so, then it would imply less degree of f - contraction in the seventh period, this being important for work on chemical identification of elements beyond kurchatovium.

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Table I

Series No	Target	Bombarding particle (energy in MeV)	Capillary column material	Temperature °C	Number of expts	I:10 <sup>-17</sup>	N <sub>0</sub>	N	N <sub>0</sub> /N
I*	242Pu	22Ne /119-117/	glass	300	2	8.6	172	13/2	8
				350					
II	242Pu	22Ne /113-118/	glass	300	8	12.1	242	28/10	12
				350					
III	242Pu	22Ne /116-118/	KCl	300	1	3.4	68	0	0
				350					
IV	242Pu	22Ne /129/	glass	300	2	6.0	12	2/1	16
	242Pu	18O /88/	glass	300	1	2.6	0	1/1	-
	243Am	18O /92-94/	glass	300	2	6.0	0	1	-

\* Chloride carriers (mm of Hg): NbCl<sub>5</sub>(0.15) + ZrCl<sub>4</sub>(0.05)

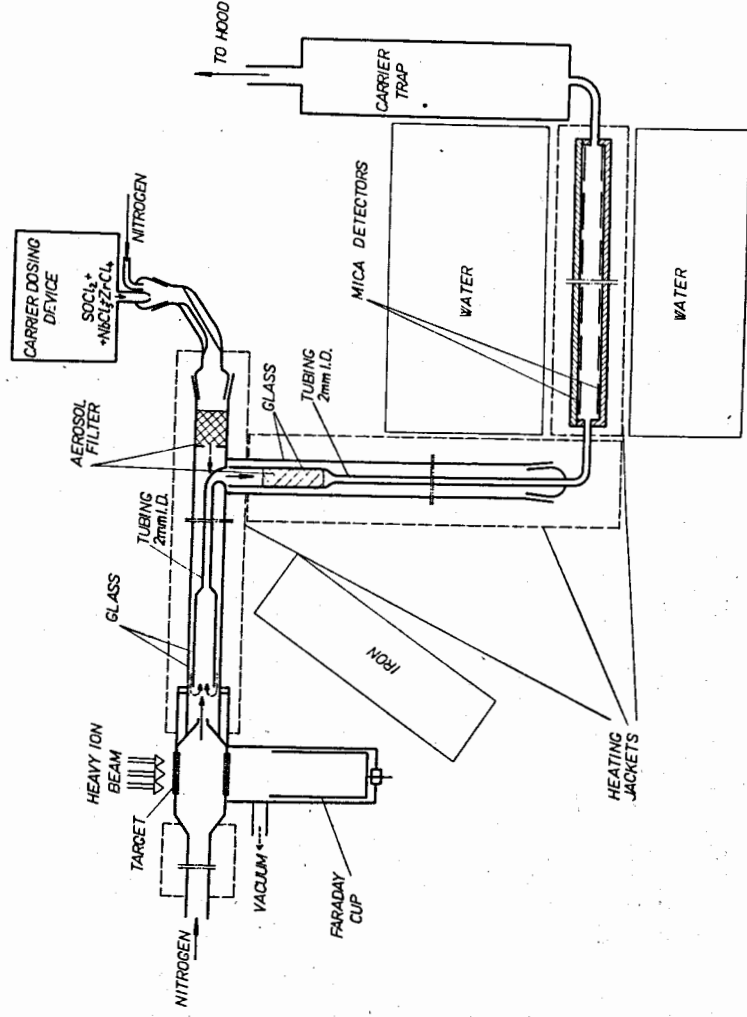


Fig. 1. Diagram of the experimental apparatus on the external beam of the cyclotron U-300.

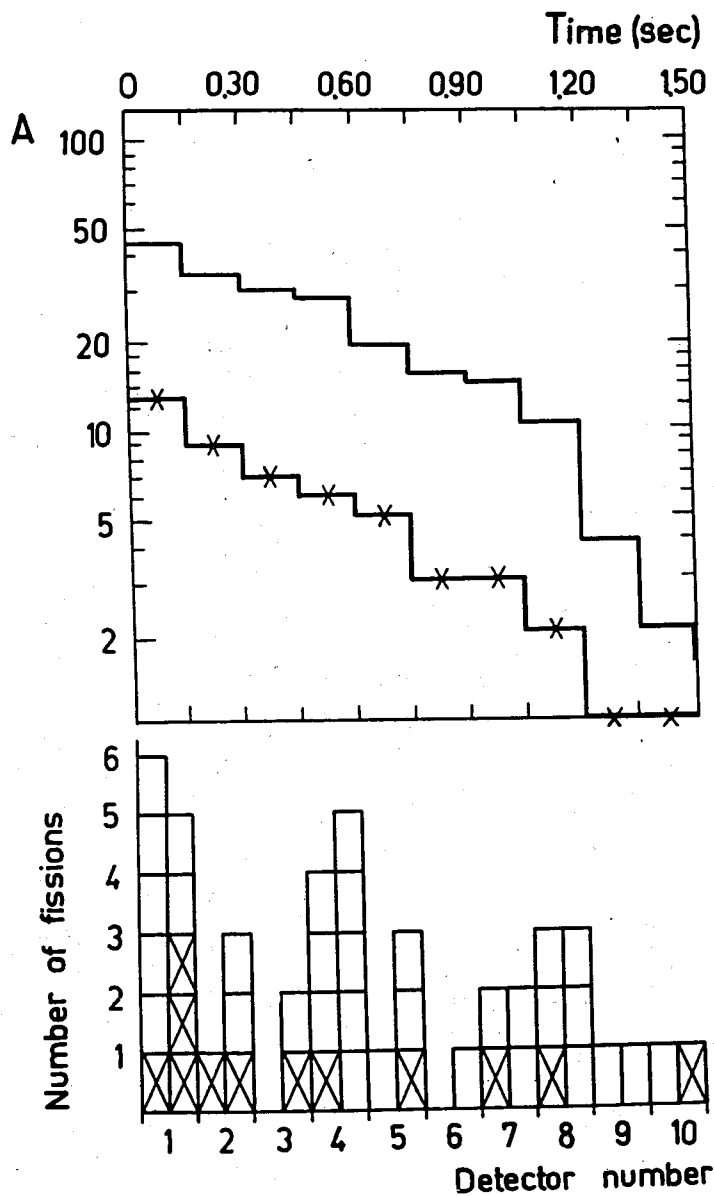


Fig.2. Bottom: The distribution along the detectors of fission events, observed in experiments of series II.  
 Top: The "decay curve" of the kurchatovium activity.  
 Crosses mark the fission events with both fragments detected and the corresponding decay curve.

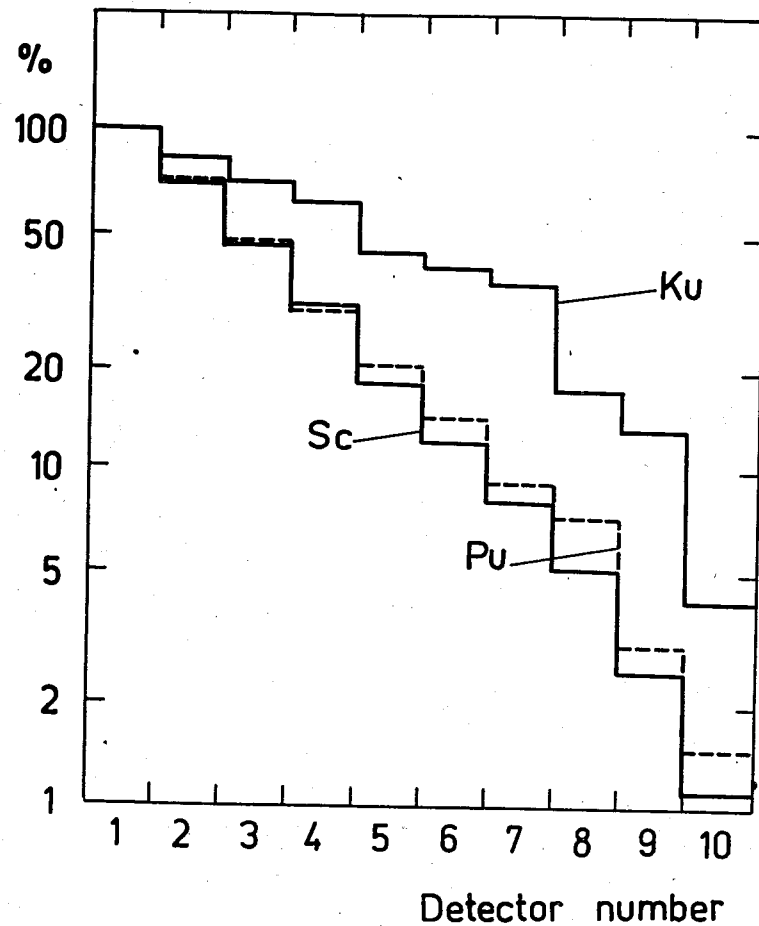


Fig.3. The percent distribution of fission events along the detectors as compared with that of plutonium and scandium isotopes.

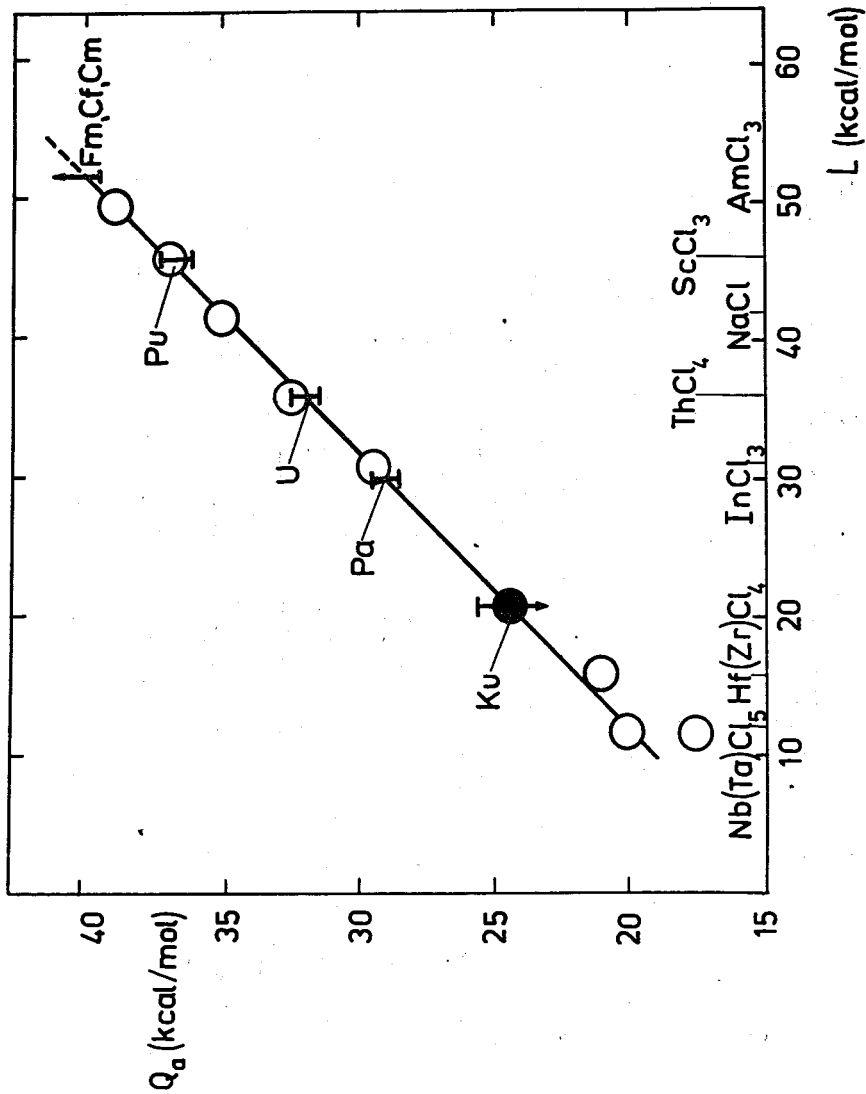


Fig.4. The experimental correlation between heat of adsorption and heat of vaporization of chlorides.