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RECENT TRANSURANIUM ELEMENT RESEARCH AT BERKELEY

Albert Ghiorso

October 1967

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Lawrence Radiation Laboratory
University of California
Berkeley, California

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Mr. Chairman, most distinguished ladies and gentlemen, it is indeed a great privilege for me to appear at this assemblage honoring Marie Sklodowska-Curie. In the early stages of my scientific career, some twenty-five years ago, I developed an enormous respect for the pioneers in the field of the radio-elements. It was my task to repeat some of the early measurements using techniques far more advanced than those available to Marie Curie and her collaborators. It became clear to me that these people of necessity had substituted skill and ingenuity for sophisticated equipment. The speed with which they proceeded in making their great discoveries was really quite remarkable.

I was fortunate to be one of Dr. Seaborg's collaborators in the discovery of element 96 which we named curium in honor of Marie and Pierre Curie. So rapid is the scientific pace in modern times that it was only some 13 years before other isotopes of curium were being used in bombardments to make the much heavier element 102 and today I want to start out by reviewing briefly what we know about nobelium some ten years later. For a detailed discussion of the complicated history of this interesting element I refer you to a recent article in Physics Today.¹

The first successful work on element 102 was accomplished early in 1958 by the method illustrated in Fig. 1. In this experiment, which was

* This work was done under the auspices of the U. S. Atomic Energy Commission.

performed at Berkeley with the Heavy Ion Linear Accelerator (HILAC), the object was to demonstrate the existence of element 102 (produced by the bombardment of curium by carbon ions) by the indirect method of milking fermium daughter nuclei produced by alpha decay of element 102. This work was successful² but, as became apparent years later, it was confused by the fact that not one but two isotopes of element 102 were produced, 3-second mass 252 and 50-second mass 254. In the strictly chemical experiments wherein the daughter recoils were analyzed chemically, the isotope ^{250}Fm was found thus establishing its alpha-particle emitting parent to be $^{254}_{102}$. In the physical experiments, wherein the daughter nuclei were analyzed as to their relative position on the daughter-catching foil to determine the parent half-life, a value of 3 seconds was found. This value was accordingly assigned to mass 254 but in reality this half-life was that of $^{252}_{102}$. It appears now that the granddaughter of mass 252, ^{244}Cf , had been confused with ^{250}Fm because of a similarity in half-life and energy.

In 1959 a 3-second activity was found in experiments in which a Frisch-grid chamber was used to analyze atoms made in these reactions. An alpha-particle energy of 8.3 MeV and a spontaneous-fission branching ratio of about 30% was determined. Since the half-life corresponded to that of the previous experiment it was natural to assume that the 8.3 MeV activity was due to mass 254.

Further work probably would have cleared up the mass 252, 254 discrepancy but a few months after these experiments were initiated we had a very terrifying experience which strongly inhibited us for a long time in the use of highly radioactive materials such as ^{244}Cm . The basic method of these grid chamber experiments was as follows. A tape transport was used to

shuttle the transmuted atoms from a target chamber filled with helium to a grid chamber filled with an argon-methane mixture. Both chambers operated at atmospheric pressure and were essentially open to the laboratory atmosphere. The target chamber was isolated from the vacuum of the Heavy Ion Linear Accelerator, which produced the carbon projectiles, by a double window of thin nickel foils cooled by a flowing stream of helium. Because of a malfunction, the pressure in the volume between the thin windows suddenly increased greatly and ruptured the window foil immediately adjacent to the curium target. This explosion, for such it was, broke the target into micron-size particles and expelled most of the activity (10^{12} alpha particles per minute) into the laboratory! I do not think that even Mme. Curie ever had to contend with spreading as much as a gram of radium around her laboratory for this is the approximate equivalent which, within a few minutes, covered the interior of the HILAC building. Fortunately, no one was seriously exposed to the radioactivity but it was several weeks before we were able to operate the accelerator again.

We turned our attention to the use of the small amounts of californium, which had become available through reactor bombardments of curium, to produce element 103. These experiments were successful³ finally in 1961 in producing an 8-sec 8.6 MeV alpha-particle activity which we deduced was probably mass 257 although masses 258 and 259 were not excluded. As a by-product of these experiments we also uncovered a 15-sec 8.2 MeV activity which we thought might be $^{255}_{102}$. It now turns out that in reality it was $^{257}_{102}$.

We did not pursue any further the work on element 102 until last year
in the USSR
when the final results of research at Dubna/, performed in the period between

1962-1966 under the direction of my good friend, G. N. Flerov, were published showing several important discrepancies with our 1958-1961 results. Responding to this challenge, T. Sikkeland, M. Nurmia and the author repeated the old experiments and substantially extended them by bombarding separated isotopes of curium with ^{12}C and ^{13}C ions in a new apparatus.⁴

The experimental equipment is shown in Figs. 2 and 3. The recoiling transmuted atoms are stopped within a small helium-filled chamber at 760 torr and carried through an orifice into a low-pressure region (about 0.1 torr) by the rapidly flowing stream of helium. The atoms travel a few mm and impinge upon the periphery of a wheel which is periodically rotated in digital fashion to expose the transmutation products successively to each of four solid state alpha-particle detectors. The half-lives and energies of the activities can be measured with good precision. A variation of this technique which was developed in our laboratory some years ago was also used by Flerov's groups in their experiments with element 102.

Using this system we have been able to iron out the apparent discrepancies and we now find reasonable agreement with the Dubna experimenters where there is an opportunity to compare results. Table 1 presents a comparison of the results obtained by the two laboratories. We have produced isotopes of element 102 from mass 251 through 257 and have set meaningful limits on the properties of mass 258. We expect to be able soon to produce $^{259}_{102}$ which may have a half-life as long as an hour and make it possible to do complicated kinds of chemistry with element 102. After clearing up the confused situation we decided that it would be best if we did not give the element a new name and consequently we have suggested that the original hastily-given one of

nobelium be retained. It is a very honorable and worthy name for an element to bear.

I would now like to report on the outcome of some very recent experiments on the solution chemistry of nobelium which have a very important bearing on the Seaborg concept of the heavy elements as an actinide series homologous to the rare earth lanthanide elements. This work, which is still in progress, is by J. Maly (on leave from Czechoslovakia), T. Sikkeland, R. Silva and the author. We have made use of the 3-minute ^{255}No isotope produced by the reaction $^{244}\text{Pu}(^{16}_0\text{n})^{255}\text{No}$. This nuclide decays by the emission of 8.1 MeV alpha particles and these are measured in a Frisch grid chamber to obtain the highest counting yield. With an $800\ \mu\text{g}/\text{cm}^2$ target bombarded by a $2\ \mu\text{a}$ oxygen beam for 10 minutes typically at the end of irradiation we have had about 20 countable atoms present and the losses undergone by nuclear decay and chemical yield have resulted in the final detection of 1-3 countable atoms per experiment. This number is quite sufficient for certain types of chemistry if many experiments can be performed so that the results are additive.

The point of the experiments that were performed may be seen from the following simplified picture of the relative tendency of the actinide elements to have +2 valence states. About fifteen years ago Seaborg suggested that the actinide elements should show an increasing tendency to form a +2 state as the atomic number is increased, reaching a peak at element 102 and then falling to zero at element 103. A few months ago in our laboratory it was found that mendelevium could be reduced to the +2 state without difficulty^{5,6} and that under certain circumstances a +2 state could be observed in Cf, Es, and Fm.⁷ It thus seemed that nobelium should have an observable +2 state but

I do not think anyone was prepared for the finding that it is a very stable +2 state. It turned out to be harder to oxidize it to the +3 state.

In the first experiments it was found that nobelium was electro-deposited from NH_4Cl solution only with a very low yield, comparable to radium which has a yield about a factor of 5 below that for Am^{+3} . The manner in which this type of plating proceeds is connected with the circumstance that the pH is high at the cathode and the yield of the tracers is approximately inversely proportional to the solubilities of their hydroxides. This experiment suggested that the hydroxide of No behaves like that of Ra^{+2} .

In the next experiments attempts were made to elute the nobelium from a Dowex 50 cation-exchange resin column with an ammonium α -hydroxyisobutyrate solution. It was found that it definitely eluted after Am--in a total of about 20 experiments no α counts of No were recorded in the pre-Am fraction where 50 events should have been observed in that fraction if No had been in its +3 state. Attempts to oxidize the No to the +3 state by the use of Ce^{+4} in order to elute it early from the column failed because Ce^{+4} and thus probably No^{+3} were readily reduced by the eluant.

The third set of experiments was just performed last week and the data are tentative. A short precipitation column consisting of BaSO_4 powder was used with H_2SO_4 as eluant in oxidation experiments. It was found that without oxidation No and Ra were washed slightly later than Am from the column. After oxidation with Ce^{+4} the No was eluted together with Am and slightly ahead of Ra. In the latter experiment one cannot tell the difference between the +3 and +4 states of No.

These preliminary experiments strongly suggest that the +2 state is

the most stable one of nobelium in solution and that it has very unusual and important chemical properties. In further experiments we hope to increase the yield of ^{255}No by bombarding a new target of ^{246}Cm with more intense beams of ^{13}C ions and foresee that perhaps as many as 100 countable atoms might be made in each experiment.

Returning to nuclear physics I would like to discuss some very recent experiments extending our knowledge of the systematic variations in the nuclear properties of the heaviest elements. Prompted by our recent confirmation of the surprisingly high spontaneous fission branching of ^{252}No we have searched for ^{244}Fm with the expectation that it might have an even shorter half-life for this mode of decay. Bombardments of ^{233}U with ^{16}O ions were soon successful⁸ and showed the existence of a 3.3 ms spontaneous fission activity which we believe to be ^{244}Fm . A simple method was used in these experiments. The transmuted atoms were caught directly in the surface of a swiftly rotating wheel and the detection of subsequent short-lived fission decay was made by examining strips of mica placed next to the wheel. A suitable etching process enormously amplifies the original fission tracks and makes them easily visible under a microscope. Figure 4 is a composite decay curve from all the experiments and Fig. 5 is the excitation function for this activity. The theoretical curve is fitted to the data at the peak of the function. We have felt that it is very important to determine the fission half-lives of the light Fm isotopes to enhance our picture of the systematic variations in the heavy element region. Accordingly we have also determined the fission branching of ^{246}Fm and ^{248}Fm for the first time.⁸

When this and other information is added to the known correlations we

see the consistent picture illustrated in Figs. 6, 7 and 8 in which are plotted alpha-particle energy, partial alpha half-life, and partial spontaneous fission half-life versus neutron number. On the basis of these empirical correlations I would like to comment on the characteristics that are presently claimed for the activity which has been designated as $^{260}_{104}$. In extremely difficult experiments performed in the years 1964-1966 by the Flerov groups at Dubna an 0.3-sec spontaneous fission activity was discovered⁹ which was thought to be due to element 104. From the energetics of the $^{22}_{\text{Ne}}$ reactions possible with the $^{242}_{\text{Pu}}$ target material the experimenters decided that mass 260 was the most suitable choice to explain their findings. Unfortunately, no alpha-particle branching was observable down to a level equal to that of the fission decay. From our empirical fission half-life correlation it would appear that the half-life is much too long by orders of magnitude to be due to $^{260}_{104}$. Since, however, an odd neutron isotope of element 104 would be hindered for this mode of decay by five or more orders of magnitude $^{259}_{104}$ or $^{261}_{104}$ might be responsible. From the alpha half-life correlation the limit on the half-life would appear to be much too long either for $^{259}_{104}$ or $^{260}_{104}$ but might be consistent with the value for $^{261}_{104}$.

A further bit of information due to Sikkeland is provided in Fig. 9 wherein is plotted the ratio of Γ_n/Γ_f vs neutron number.¹⁰ This parameter represents the average competition between neutron emission and fission which takes place in a compound nucleus reaction at each step in a nuclear cascade when neutrons are emitted following excitation by the amalgamation of the heavy particle projectile with the target nucleus. Whereas all of the other experimental data fit the family of curves quite well, including

the three new points for fermium isotopes, the value calculated assuming a $(^{22}\text{Ne}, 4n)$ reaction to produce $^{260}_{104}\text{Fm}$ is much lower than the expected one.

In the same figure we have also indicated the average Γ_n/Γ_f values for the cases assuming that the spontaneous fission activity had been produced in a $(^{22}\text{Ne}, 5n)^{259}_{104}\text{Fm}$ or a $(^{22}\text{Ne}, 3n)^{261}_{104}\text{Fm}$ reaction. It appears that the latter gives the best fit. It must, however, be pointed out that such an extrapolation of Γ_n/Γ_f values to unknown nuclides past the 152 neutron subshell is quite uncertain.

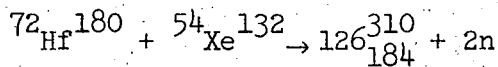
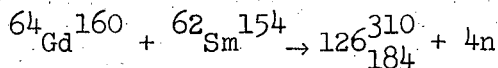
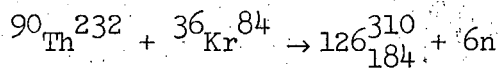
From all the evidence given above it would seem that the 0.3 sec fission activity might well be due to $^{261}_{104}\text{Fm}$. There is also the remote possibility that the activity is not due to element 104 at all but is a neutron-heavy isomer of an element of lower atomic number such as Np or Pu which also have very volatile chlorides.

We will soon make some attempts to corroborate and extend the information concerning isotopes of element 104. We now have available in our laboratory 55 micrograms of ^{249}Cf . This nuclide was obtained by milking it from several hundred micrograms of ^{249}Bk which has been obtained in quantity for the first time by intense irradiation of curium in the High Flux Isotope Reactor at the Oak Ridge National Laboratory. Bombardment of this target material with ^{12}C and ^{13}C ions should produce isotopes of element 104 with masses 257-259. The use of ^{252}Cf as a target material will produce masses 260-262. The use of ^{14}N and ^{15}N and ^{16}O and ^{18}O will produce isotopes of element 105 and 106 but possibly at such a low rate that the effort will not be deemed worthwhile in our laboratory.

The greatest obstacle to the production of elements with ever higher

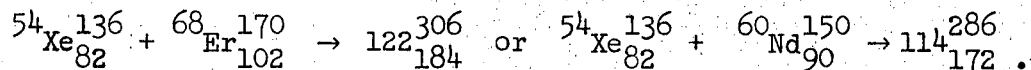
atomic number is set by their production cross sections. The very short half-lives likely to be encountered can be coped with by modern techniques, but soon the production rates will fall below a profitable level. This comes about because as the atomic number is increased the fission barrier is lowered and consequently the Γ_n/Γ_f ratio is decreased as shown in Fig. 9; the end product soon becomes almost entirely that of fission. There is however the distinct possibility of "leap-frogging" this difficulty.

We know of no fundamental reason why there should be no magic numbers after $Z = 82$ and $N = 126$. A crude shell model calculation suggests that 126 protons and 184 neutrons would be the next stabilizing combination.¹¹ With this view the closed shell at this point would be a very strong one and would lead to a peak in the fission barrier as high as 12 MeV on this island of stability.¹¹ This means that the spontaneous fission half-lives in this region would be very long. Alpha half-lives are more uncertain but it seems that there is a region where they might be as long as years. Figure 10 is a contour chart of stability developed by Sikkeland which mirrors this particular closed shell hypothesis. Examples of fusion reactions which might produce the closed shell nucleus ${}_{126}^{310}\text{184}$ are the following:



In the last few years there have been closer looks taken at the shell

problem by several physicists. They suggest that perhaps the next proton shell is more likely to be at 114 protons.¹² The neutron shell is probably still at 184.¹² It is impossible to make the nuclide with 114 protons and 184 neutrons by a fusion reaction because this nucleus is much too neutron-heavy for any possible combinations of projectile and target. The only hope would seem to be either a spallation reaction or fission of a much heavier nucleus. A reaction of the former type might be ${}^{68}_{\text{Er}}{}^{170} + {}^{68}_{\text{Er}}{}^{170} \rightarrow {}^{114}_{184}{}^{298} + (22p20n)$ where the group of 22p and 20n might be emitted as a result of fragmentation. An example of the fission reaction might be ${}^{238}_{\text{U}} + {}^{238}_{\text{U}} \rightarrow {}^{114}_{184}{}^{298} + {}^{70}_{\text{Yb}}{}^{178}$. The hope in this case is that the doubly-closed shell might be a favored fission product. Other possibilities would be to produce nuclides of either 114 protons or 184 neutrons with the hope that the stability would extend to the vicinity of the doubly-closed shell nucleus. In these cases reactions of the following type might be suitable:



Obviously, to be able to explore so many different reactions requires an accelerator which is extremely flexible. It is desirable that it should accelerate the heaviest of the elements that are available as well as the lightest elements to any energy in the range of 5 to 20 MeV per nucleon. We have designed such an all-purpose accelerator¹³ and it is presently under serious consideration for construction at Berkeley in the next few years. We have labeled this machine the "Omnitron" because it does fulfill all of these considerations and more.

The fundamental limitation of resonant types of accelerators for very

heavy ions is found to be in the ion source. For nonrelativistic velocities, which is the aim here, the velocity is simply $v = \frac{q}{m} \cdot B\rho$ where $B\rho$ is the magnetic rigidity of the ion to be accelerated. Thus, as the mass m is increased the charge q on the ion must increase in the same ratio to maintain a given velocity if the maximum value of $B\rho$ available in a given system is to be used. Unfortunately, as m is increased it becomes ever more difficult to strip enough electrons from the atom. Figure 11 indicates typical intensity distributions available from our HILAC ion source for various elements. Typically, charge-to-mass ratios as high as 0.15 are needed to accelerate ions to the required velocity for our purposes either in large cyclotrons or in existing linear accelerators. The Omnitron avoids this problem by using q/m ratios as low as 0.05 but, of course, must provide a large bending radius. The system that we have conceived is shown in Fig. 12 and makes use of a strong-focussing alternating gradient 60 Hz synchrotron concentric with a dc storage ring of similar design. The diameter of the system is approximately 100 feet. Notice that two dc injectors are used to allow for sequential acceleration of two different ions for concurrent operation. This feature is especially desirable for an expensive machine of this sort so that the system can be made available to more than one experimenter on a time-sharing basis. Figure 13 indicates the two important modes of operation that are available from the Omnitron. The first makes it possible to convert the fractional microsecond beam bursts available from the accelerating ring at 60 Hz into essentially dc beam by making use of the storage ring to extend the beam spill. This beam will have a very small energy spread and a high brightness. The second mode permits the acceleration of particles to very high energies such

as 400 MeV per nucleon by a two-step process. The first step involves acceleration to energies of 10-30 MeV per nucleon at a low charge-state, storage for a half cycle until the magnetic field has returned to its minimum value and then reinjection through a stripper foil to gain a high charge-state and thus allow a second step of acceleration to a much higher energy. Protons can be accelerated in a single cycle to 1.7 GeV.

The main objective in accelerating heavy ions such as neon or argon to high energies is to make possible their use in biomedical research and medical therapy. These relatively heavy particles will be extremely important in this work because of the large amounts of energy that can be lodged per particle into organic materials by virtue of the large equivalent Bragg effect observed at the end of the range. Since only very small beams are necessary for this use it should be possible to make them available a large percentage of the time at a low duty cycle.

The Omnitron is an extremely flexible system and capable of fulfilling a wide variety of experimental needs. We believe that it will open up new areas of research to fruitful investigation by many groups of research workers. It is our intention that this machine will be used on an international basis and be another instrument to further the dream of Marie Sklodowska Curie that her science should serve humanity.

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March 19, 1968

ERRATUM

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Subject: UCRL-17907, "Recent Transuranium Element Research at
Berkeley," Albert Ghiorso, October 1967

Page 15. Please replace Table I by the attached table.

Table I. Production schemes and decay properties of various isotopes of element 102.^a

Isotope	Reaction used	Peak cross section ^c (10 ⁻³⁰ cm ²)	Half-life (sec)	QE (MeV) (±.02)	SF/α ratio	Reaction used	Half-life (sec)	QE (MeV) (±.03)	SF/α ratio
251 ₁₀₂	244 _{Cm} (¹² C, 5n)	0.09	0.8±0.3	8.60(80%) 8.68(20%)		Not reported			
252 ₁₀₂	244 _{Cm} (¹² C, 4n)	0.13	2.3±0.3			8.41	1/2 ^d	239 _{Pu} (¹⁸ O, 5n)	4.5±1.5
	244 _{Cm} (¹³ C, 5n)	0.096	~2.5 ^b	8.41	1/2 ^d				
253 ₁₀₂	244 _{Cm} (¹³ C, 4n)	0.29	105±20	8.01		242 _{Pu} (¹⁶ O, 5n)	95±10	8.01	
	246 _{Cm} (¹² C, 5n)	0.25	~100 ^b	8.01		239 _{Pu} (¹⁸ O, 4n)			
254 ₁₀₂	246 _{Cm} (¹² C, 4n)	0.89	55±5	8.10		243 _{Am} (¹⁵ N, 4n)	--	8.11	≤1/1800
	246 _{Cm} (¹³ C, 5n)	0.54	~50 ^b	8.10		238 _U (²² Ne, 6n)	50±10		
	244 _{Cm} (¹³ C, 3n)	0.096	~50 ^b	8.10		242 _{Pu} (¹⁶ O, 4n)	75±15		
255 ₁₀₂	246 _{Cm} (¹³ C, 4n)	0.47	185±20	8.11		238 _U (²² Ne, 5n)	~120	8.08	
	248 _{Cm} (¹² C, 5n)	0.38	~180 ^b	8.11		242 _{Pu} (¹⁸ O, 5n)	180±10	8.09	
256 ₁₀₂	248 _{Cm} (¹² C, 4n)	0.74	2.9±0.5	8.43	~1/400 ^d	238 _U (²² Ne, 4n)	6±2	8.41	1/200
	248 _{Cm} (¹³ C, 5n)	0.75	3.2±0.2	8.43		242 _{Pu} (¹⁸ O, 4n)	9±3	8.42	
	246 _{Cm} (¹³ C, 3n)	0.09		8.43					
257 ₁₀₂	248 _{Cm} (¹³ C, 4n)	1.1	23±2	8.23(50%) 8.27(50%)		Not reported			
	248 _{Cm} (¹² C, 3n)	0.08	~20 ^b			8.25			

^aData in columns 2-6 are from Ref. 14; those in the last four columns are taken from Ref. 15.

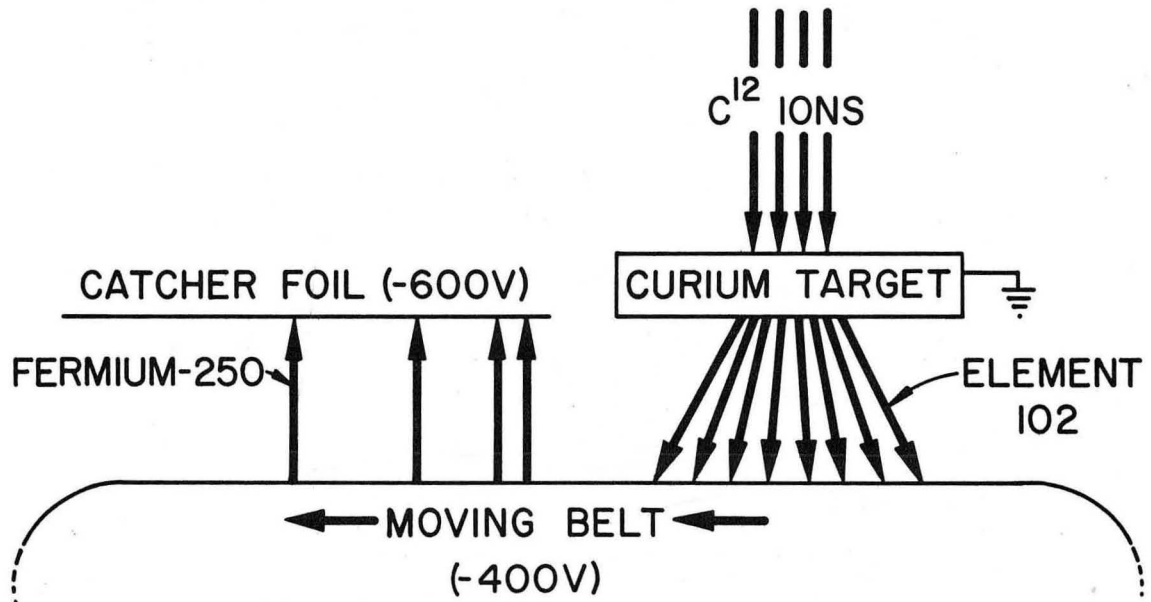
^bNo error given due to rather poor statistics.

^cThe relative values are good to within 25%, the absolute values to within a factor of two.

^dThe mass assignment of the SF emitter is not conclusive.

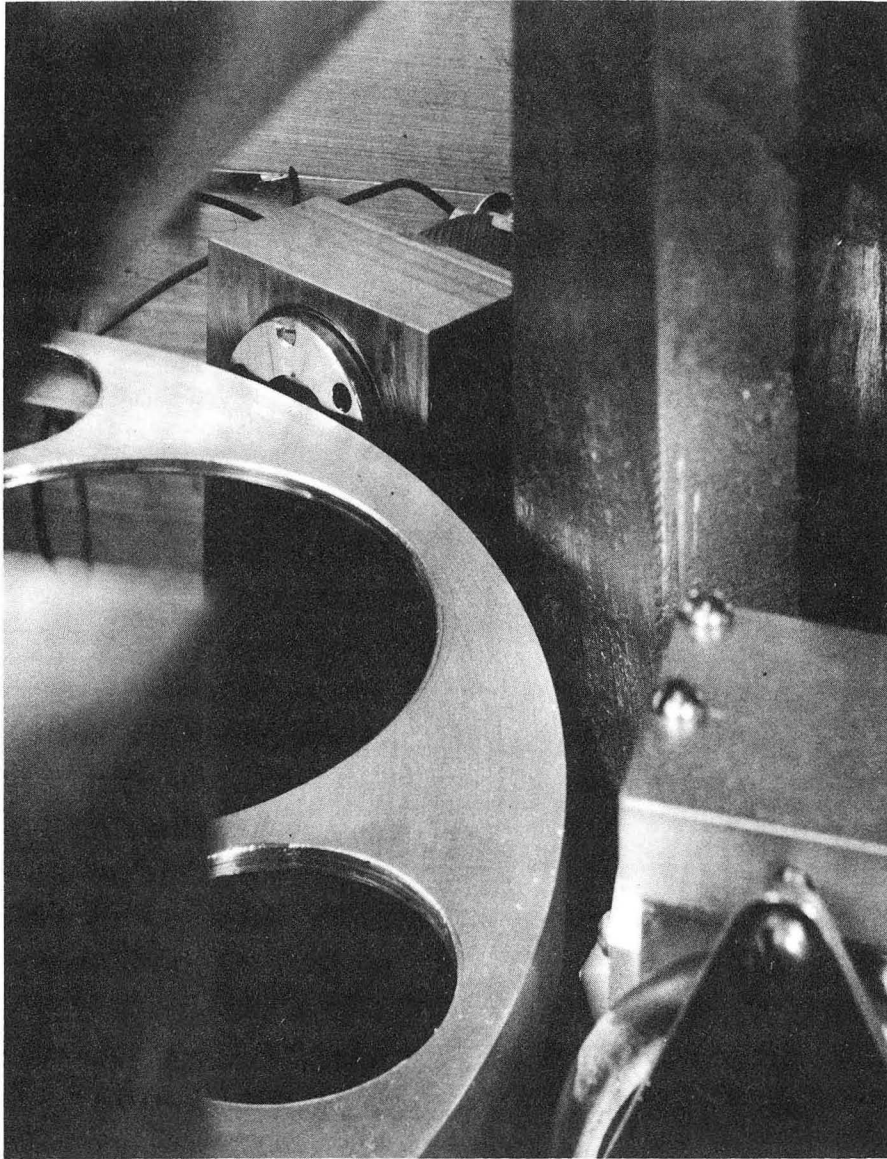
Figure Captions

- Fig. 1) Apparatus used in the Berkeley element 102 discovery experiments in 1958.
- Fig. 2) { Photographs of the apparatus used for the recent element 102
Fig. 3) { experiments.
- Fig. 4) Composite spontaneous fission decay curve from the reaction $^{233}_{92}\text{U}(^{16}_0\text{n}, 5\text{n})^{244}_{102}\text{Fm}$.
- Fig. 5) Excitation function to produce $^{244}_{102}\text{Fm}$.
- Fig. 6) Alpha particle energy vs neutron number.
- Fig. 7) Partial alpha half-life vs neutron number.
- Fig. 8) Partial spontaneous fission half-life vs neutron number.
- Fig. 9) Γ_n/Γ_f vs neutron number.
- Fig.10) Hypothetical stability chart assuming closed shells at 126 protons and 184 neutrons. Prepared by T. Sikkeland, 1967.
- Fig.11) Ion intensity output as a function of charge state for krypton and xenon.
- Fig.12.) General layout of the Omnitron.
- Fig.13) Omnitron acceleration modes for low and high energy operation.



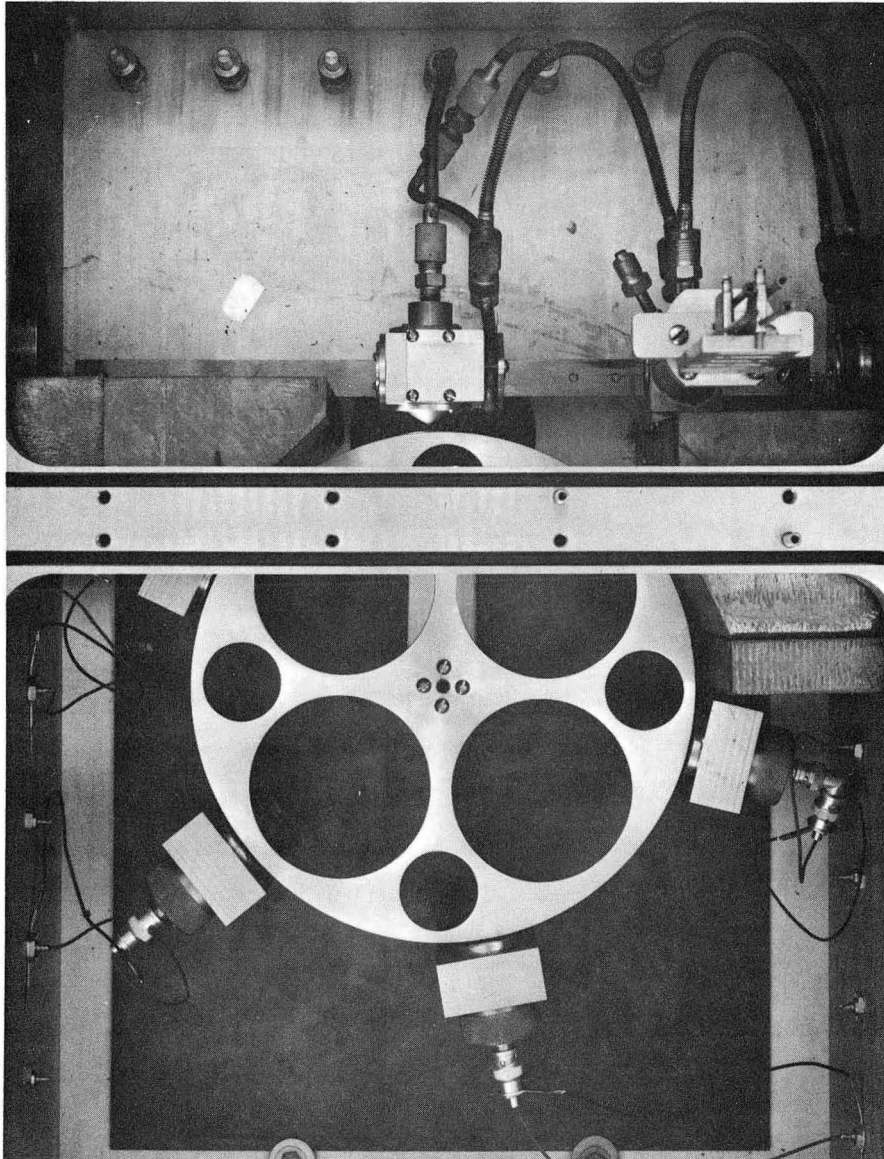
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Fig. 1



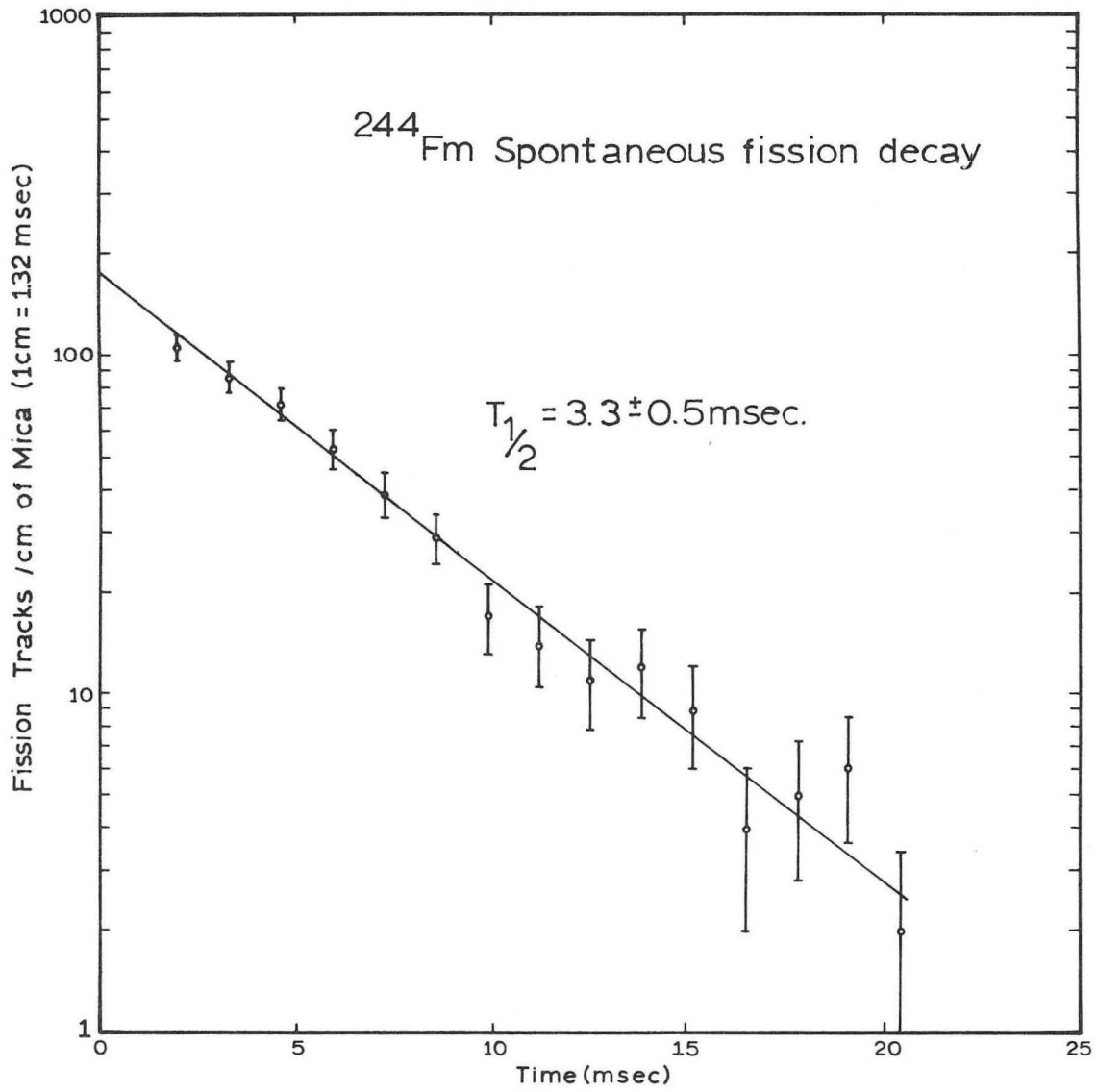
XBB 675-2515

Fig. 2



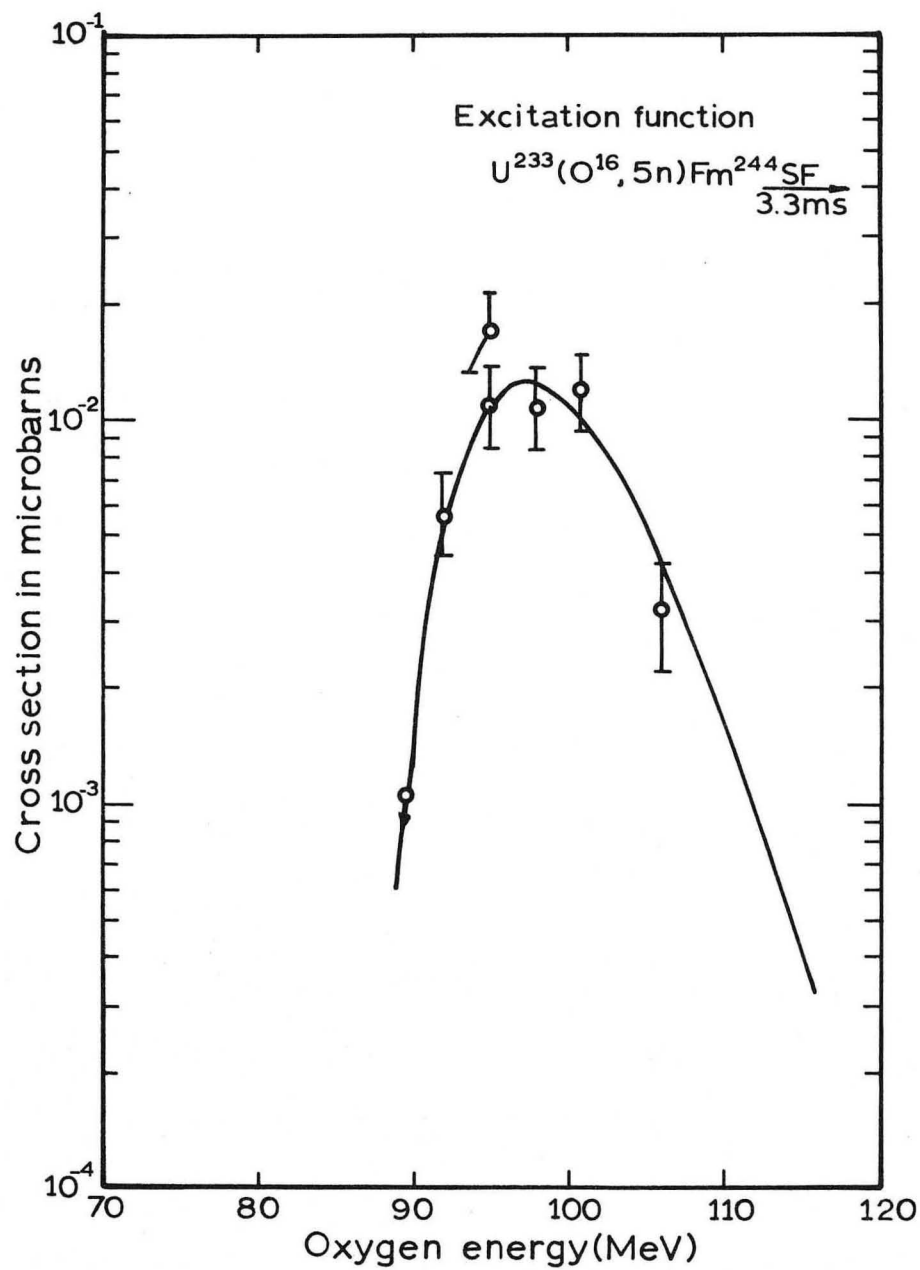
XBB 675-2514

Fig. 3



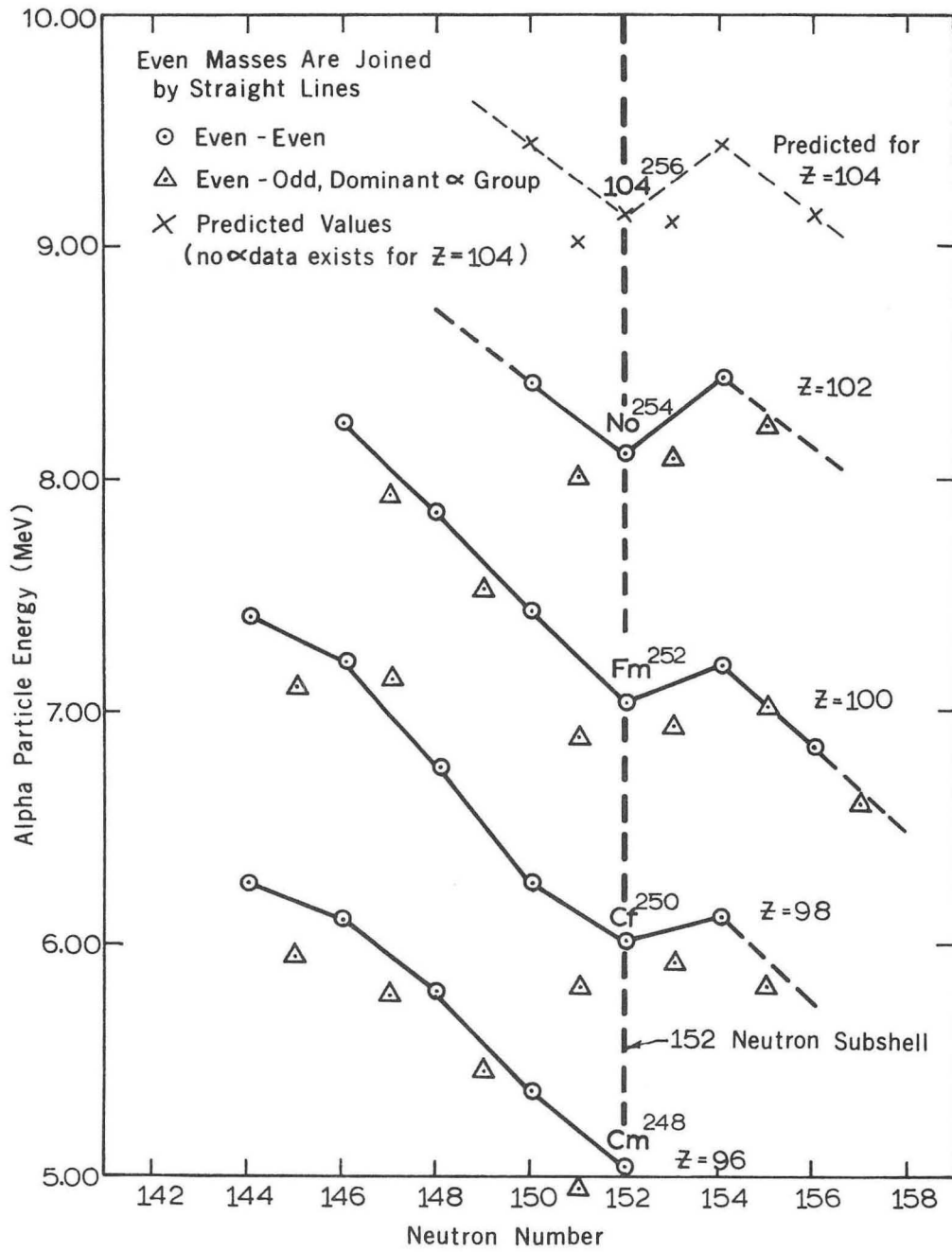
XBL 6710-2097-A

Fig. 4



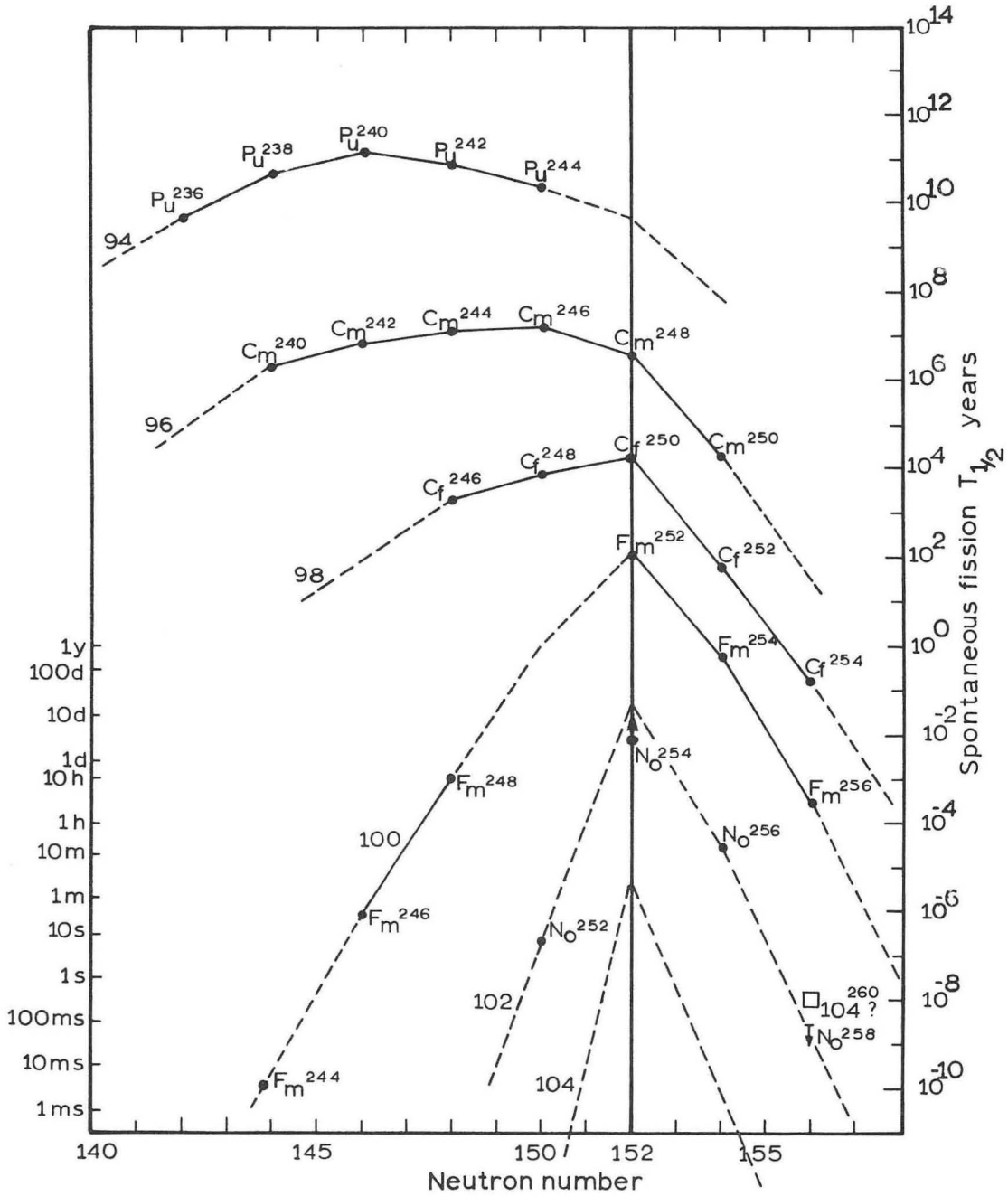
XBL 670 2096a

Fig. 5



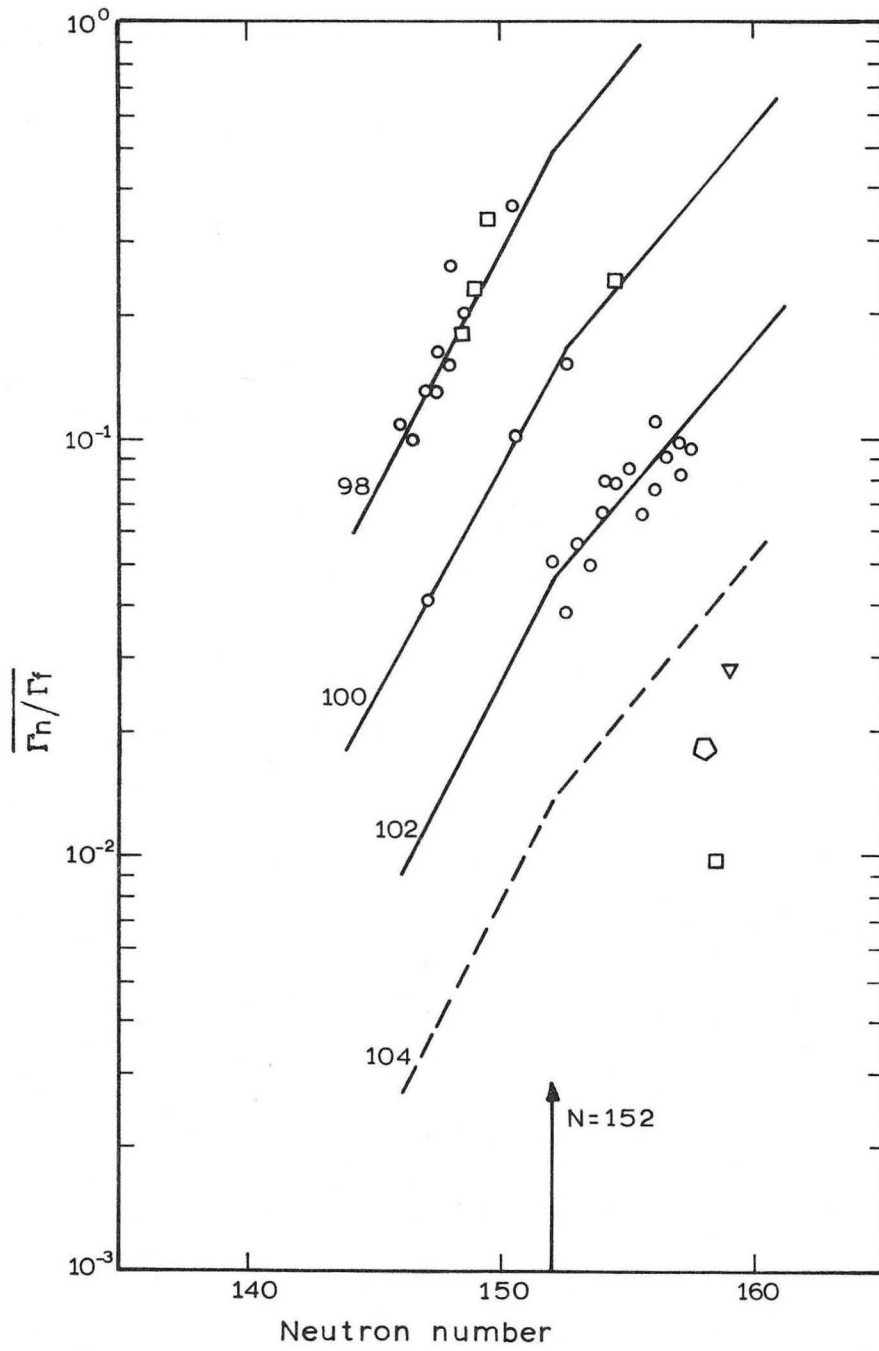
XBL6732028

Fig. 6



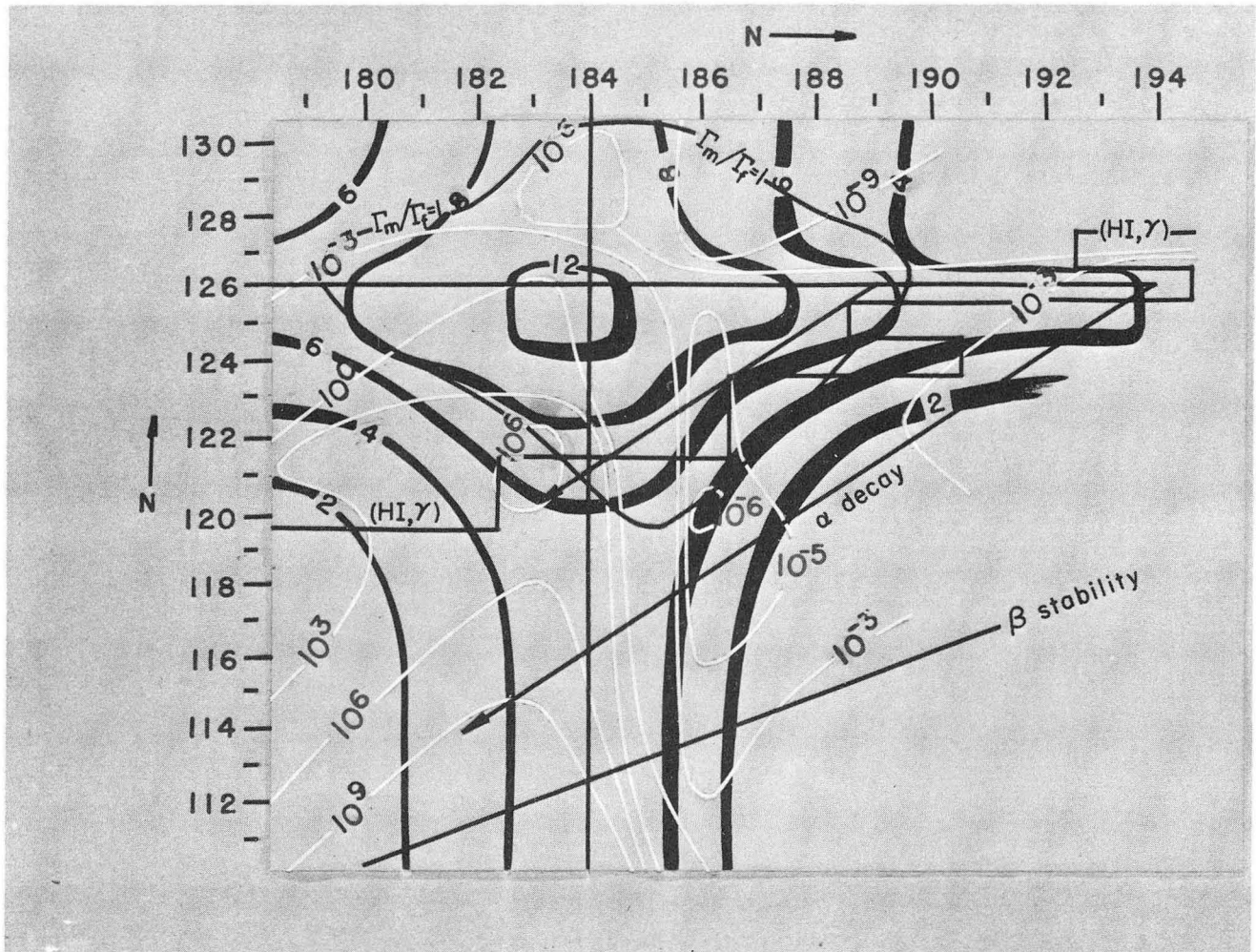
XBL 672 2016b

Fig. 8



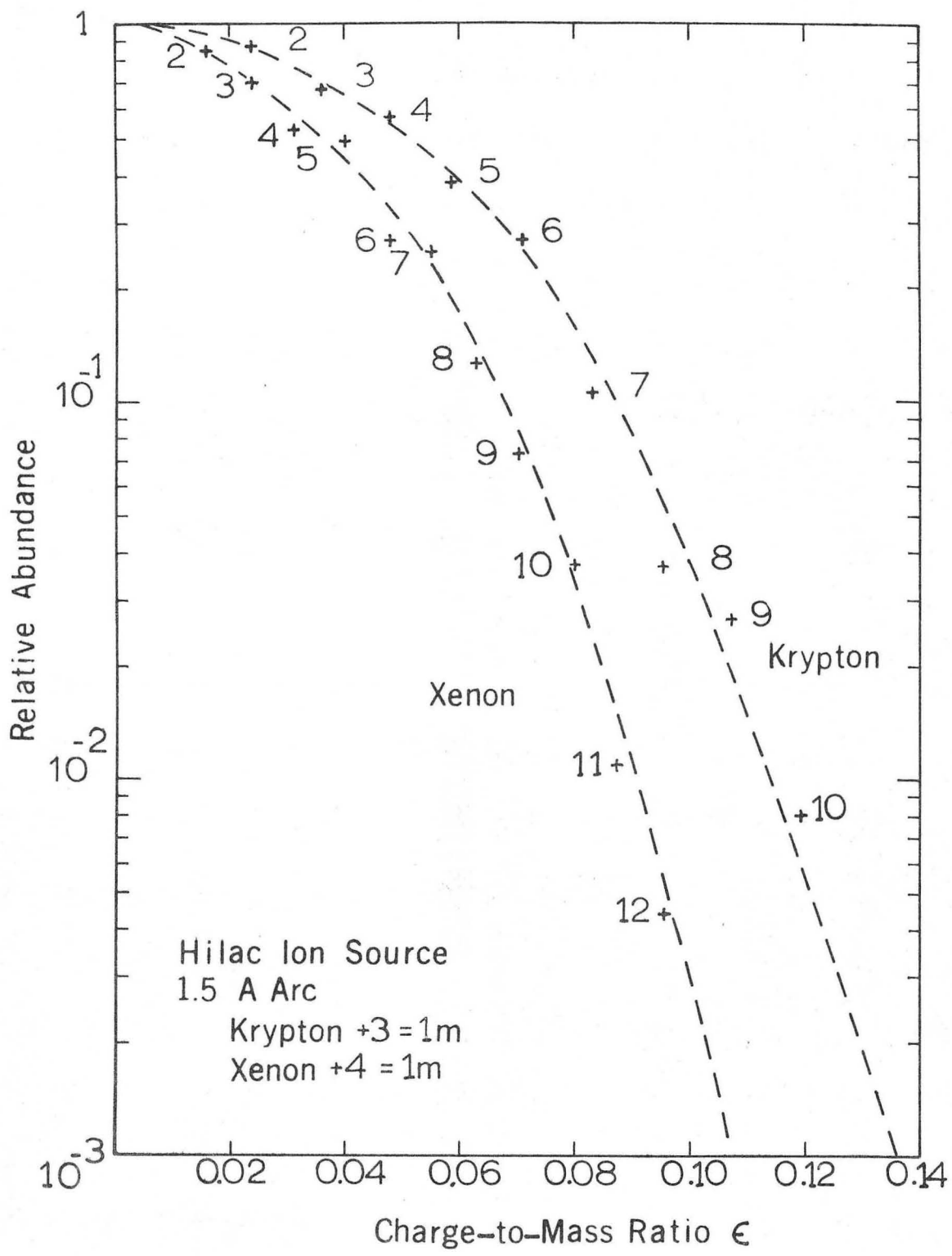
XBL 670 2095

Fig. 9



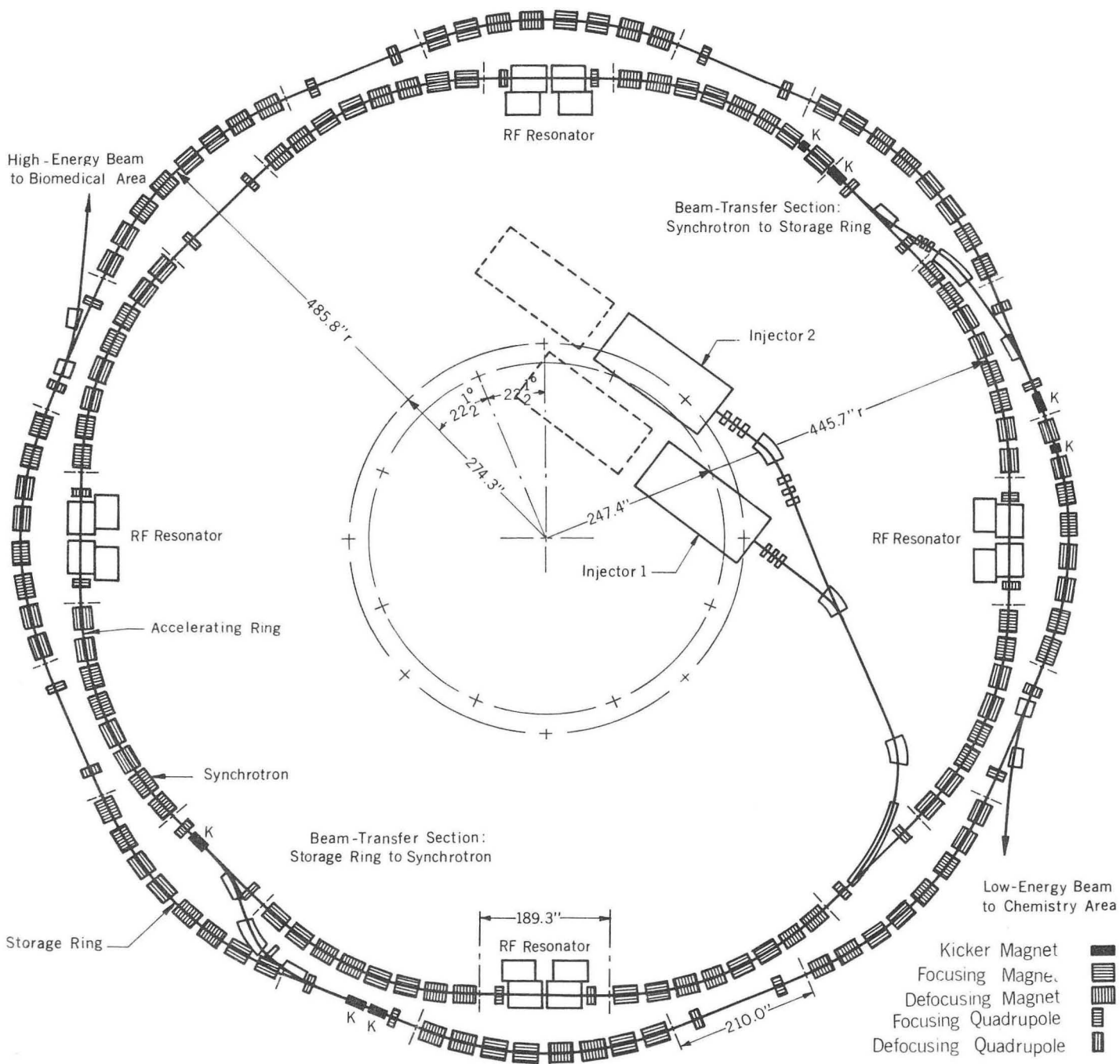
BBH 673-95

Fig. 10



Relative Abundance of Krypton and Xenon Charge States from the Hilac Ion Source

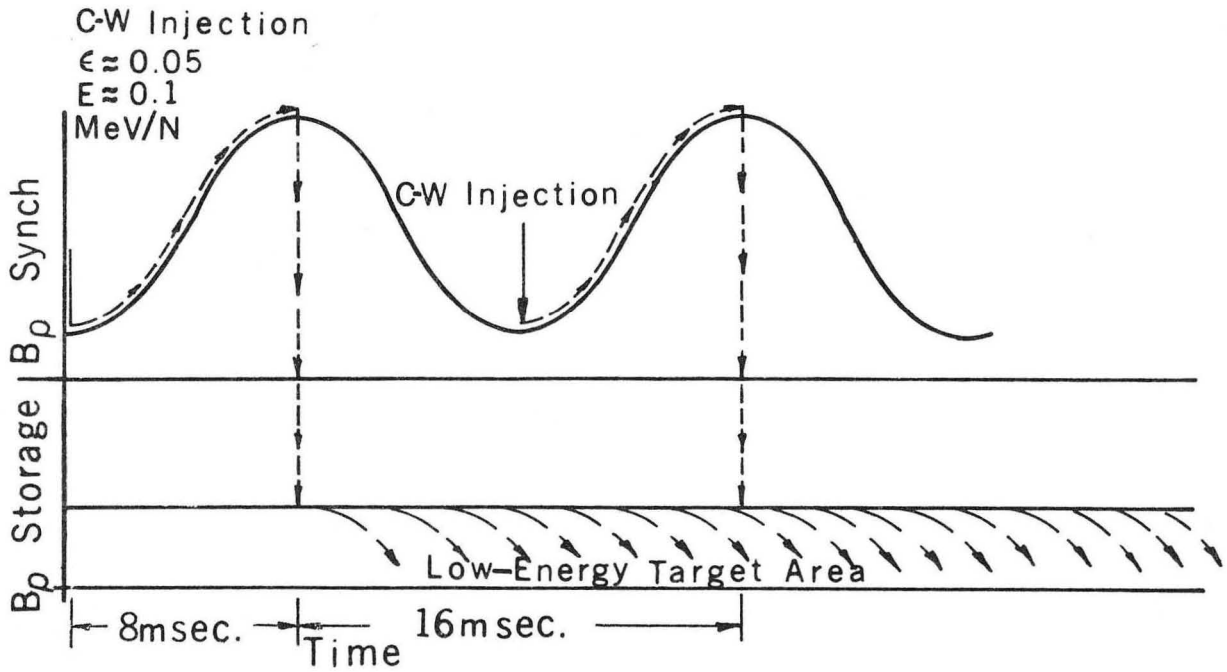
Fig. 11



General Layout of the Omnitron

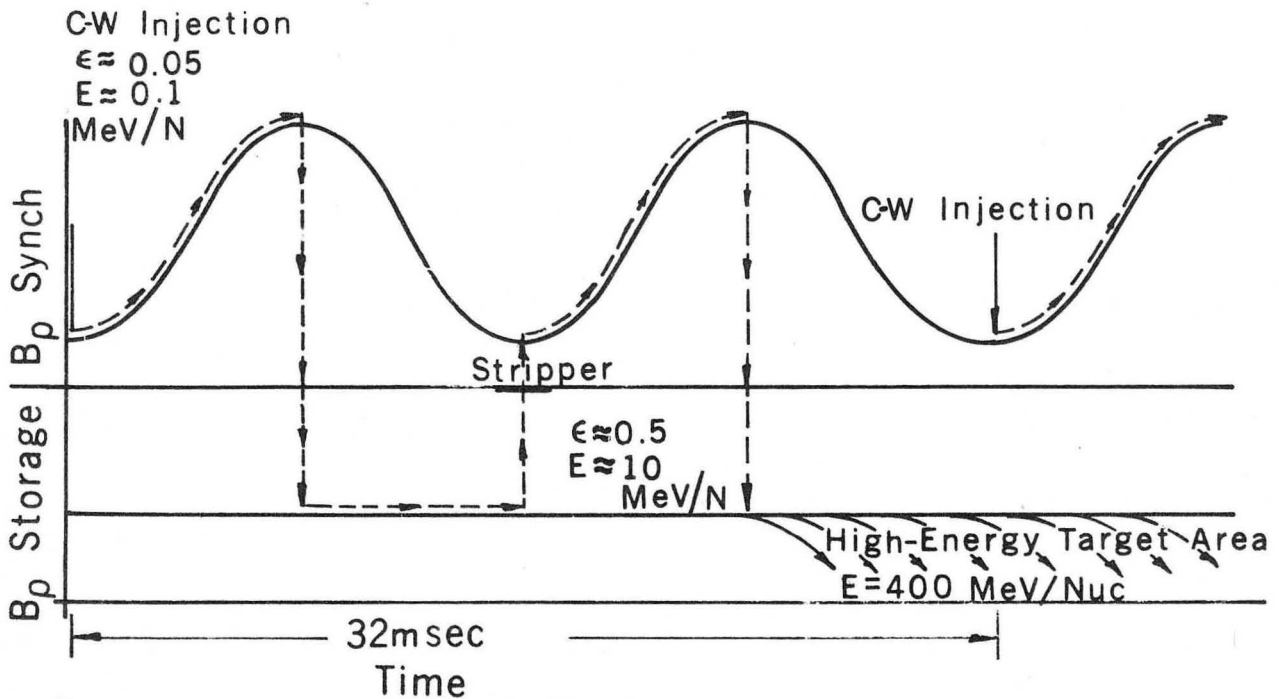
MUB 11720

Fig. 12



(a)

Single Cycle (Low Energy)



(b)

Double Cycle (High Energy)

Acceleration Cycles

MUB-11753

Fig. 13

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