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# Decay Properties of Neutron Deficient Osmium and Rhenium Isotopes. I. Decay Modes of Re$^{179}$, Re$^{180}$, Os$^{180}$, and Os$^{181}$†

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Scintillation and semiconductor detectors have been used to investigate the decay properties of neutron-deficient osmium and rhenium isotopes which were produced in proton and helium-ion bombardments of tantalum and tungsten targets. Re$^{179}$ decays by electron capture with a half-life of 19.7±0.5 min; the energies, and relative intensities of 14 $\gamma$ rays emitted in Re$^{179}$ decay are reported. The positron decay branch previously assigned to 20-hr Re$^{180}$ has been shown to occur in the decay of 13-h Re$^{182}$, and no evidence for a Re$^{180}$ isomer having a half-life longer than a few minutes was found. The existence of 2.45-min Re$^{180}$ has been confirmed; it decays predominantly by electron capture with the emission of intense 902.2- and 103.6-keV $\gamma$ rays. By establishing its genetic relationship with 2.45-min Re$^{180}$, a new isotope Os$^{180}$, decaying by electron capture with a 21.7±0.6 min half-life, has been identified. The half-life for the electron capture decay of Os$^{181}$ has been determined to be 105±3 min, in disagreement with previous values reported for this isotope. An intense 238.6-keV $\gamma$ ray is characteristic of Os$^{181}$ decay and the energies and relative intensities of other Os$^{181}$ $\gamma$ rays are reported. Reasons are advanced for believing that the Re$^{179}$, Os$^{180}$, and Os$^{181}$ activities identified here have been observed previously by other workers, but have been incorrectly assigned to Re$^{180}$, Os$^{181}$, and Os$^{183m}$, respectively.

## I. INTRODUCTION

ALTHOUGH the neutron-deficient osmium and rhenium nuclides lie at the low-mass end of the interesting rotation-vibration transition region between highly deformed and spherical nuclei, their decay properties have not been well established. This has been mainly due to the extreme difficulty in preparing radiochemically pure sources; most possible methods of production yield mixtures of isotopes having complicated $\gamma$ spectra and rather similar half-lives. The excellent energy resolution which can be achieved with semiconductor detectors has now made possible reliable and detailed studies of the decay properties of these nuclides. In this paper the half-lives and principal radiations of Re$^{179}$, Re$^{180}$, Os$^{180}$, and Os$^{181}$ are reported; the results of $\gamma$-$\gamma$ coincidence measurements and proposed decay schemes for the various nuclides will be presented in forthcoming publications.[1]

## II. EXPERIMENTAL

The radioisotopes were produced in bombardments of tantalum foils and enriched tungsten isotopes with protons and helium ions from the Argonne 60-in. cyclotron and from the Oak Ridge 88-in. cyclotron (ORIC). In general, fast radiochemical separations were performed. Tungsten targets were dissolved in a 1:5 HF-HNO$_3$ mixture, osmium and rhenium carriers were added, and OsO$_4$ was distilled into ice-cold 6*M* NaOH. In a few cases where it was necessary both to know the time of chemical separation rather precisely and to start the counting as soon as possible after the separation, the OsO$_4$ distillate was collected for a fraction of a minute and the sample was counted in liquid form. However, the distillate always contained F$^{18}$ impurity arising from traces of oxygen in the target material and in general this was eliminated by precipitating OsS$_4$ from acid solution and mounting the sulphide for counting. Tantalum targets were dissolved, together with rhenium carrier, in HF containing a minimum quantity of HNO$_3$. The *p*H of the solution was adjusted to about 9 and tetraphenyl arsonium perrhenate was extracted into chloroform. The rhenium was back extracted into aqueous solution and precipitated as Re$_2$S$_7$. In cases where extremely fast chemistry was essential, the solvent extraction step was eliminated.

Gamma spectroscopy was performed using 3-in.×3-in. NaI(Tl) crystals and lithium-drifted germanium detectors in conjunction with multichannel analyzers. A number of germanium detectors with depletion volumes ranging from 1 to 3 cm$^3$ were used at different stages of the experiments; a typical detector had an energy resolution of about 5 keV for Cs$^{137}$ 662-keV $\gamma$ rays. Positrons were detected using a con-

TABLE I. Energy standards used in calibrating the $\gamma$ spectrometers

| Primary standards[a]<br>Nuclide | $E_\gamma$ (keV) | Secondary standards (see text)<br>Nuclide | $E_\gamma$ (keV) |
|---|---|---|---|
| Cd$^{109}$ | 87.7 | Re$^{182}$ | 100.1 |
| Co$^{57}$ | 122.0 | Os$^{183}$ | 114.5 |
| Co$^{57}$ | 136.4 | Os$^{182}$ | 180.2 |
| Hg$^{203}$ | 279.1 | Re$^{181}$ | 365.5 |
| Sn$^{113}$ | 393±1 | Os$^{183}$ | 382.0 |
| $\beta^+$ annihilation | 511.0 | Re$^{182}$ | 1122.0 |
| Bi$^{207}$ | 569.6 | Re$^{182}$ | 1189.3 |
| Cs$^{137}$ | 661.6 | | |
| Mn$^{54}$ | 835.5 | | |
| Y$^{88}$ | 898.2 | | |
| Bi$^{207}$ | 1063.7 | | |
| Zn$^{65}$ | 1115.6 | | |
| Co$^{60}$ | 1173.2 | | |
| Co$^{60}$ | 1332.5 | | |

[a] Based on information in the *Nuclear Data Sheets*, edited by K. Way *et al.* (Printing and Publishing Offices National Academy of Sciences–National Research Council, Washington 25, D. C., 1961–1965), and on recently published data.

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† Supported in part by the U. S. Atomic Energy Commission.

[1] K. J. Hofstetter and P. J. Daly (to be published).

ventional two-crystal coincidence spectrometer having a resolving time ($2\tau$) of 70 nsec. Standard sources were used for energy calibrations and for determining the photopeak efficiencies of the various detectors used. The primary energy standards used most frequently are listed in Table I. This table also lists a number of intense $\gamma$ rays of longer lived osmium and rhenium isotopes, the energies of which have been established ($\pm$0.2 keV) by at least two independent groups of workers. These $\gamma$ rays were used as secondary energy standards, and they were particularly useful in interpolation and in detecting small changes in amplifier gain at high count rates. On the basis of the reproducible results obtained using different spectrometers and different sets of energy standards, the $\gamma$-ray energies given in this paper are believed to be correct within $\pm$0.4 keV except where otherwise stated.

## III. RESULTS AND CONCLUSIONS

### A. The Decay of $Re^{179}$

In bombardments of rhenium with high energy protons, Foster *et al.*[2] produced, and assigned to $Re^{180}$, a new rhenium isotope emitting 209-keV conversion electrons and decaying with a half-life of 18 min. Subsequently, Harmatz *et al.*[3] observed the decay of the same isotope and reassigned it to $Re^{179}$ on the basis of its clear cut genetic relationship with $W^{179}$. The Chart of the Nuclides[4] assigns to $Re^{179}$ a single $\gamma$ ray of 0.28 MeV, which is presumably based on interpreting the 209-keV conversion line observed by Foster *et al.* as being due to a *K* conversion electron. It is most likely[3] that this conversion line corresponds instead to *L* conversion of the 222-keV transition in $W^{179}$.

We have produced $Re^{179}$ in 68-MeV $\alpha$-particle bombardments of tantalum and have determined its half-life to be 19.7$\pm$0.5 min. The $\gamma$ spectrum is shown in Fig. 1 and the energies and relative intensities of the principal $\gamma$ rays are listed in Table II. The growth of the 222-keV $\gamma$-ray activity of $W^{179}$ was observed to follow the behavior expected for a 5-min daughter activity being fed by an initially pure 20-min parent isotope. As further confirmation of the mass assignment, the 290.0- and 429.3-keV $\gamma$-rays were observed in 32-MeV $He^3$ ion bombardments of tantalum, but not in bombardments with 26-MeV $He^3$ ions. The threshold for the reaction $Ta^{181}(He^3,5n)$ calculated using semi-empirical mass tables[5] is at about 27-MeV bombarding energy.

Recently, Bedrosyan *et al.*[6] assigned to $Re^{180}$ a 22-min rhenium activity produced in 660-MeV bombardments of gold. This group reported an intense 0.44-MeV $\gamma$ ray and some weaker $\gamma$ rays which were systematically 10-20 keV higher in energy than those listed in Table I. Almost certainly the activity observed was that of $Re^{179}$.

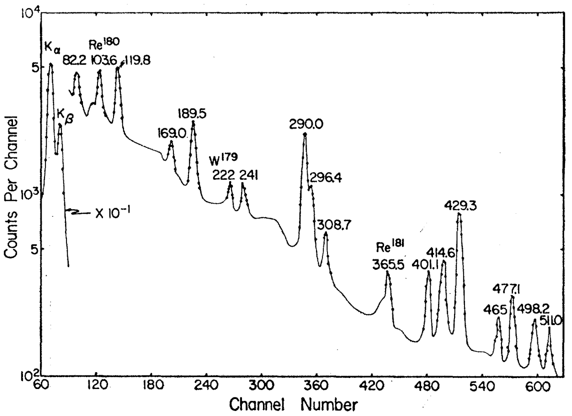

FIG. 1. The $\gamma$ spectrum of 19.7-min $Re^{179}$.

### B. The Nonexistence of 20-h $Re^{180}$

In bombardments of tungsten and rhenium with high energy protons and of enriched $W^{180}$ with 10-MeV protons, Haldar *et al.*[7] found a new Re $\beta^+$ emitter of about 20-h half-life. The $\beta^+$ endpoint energy was measured to be about 1.9 MeV and the new activity was assigned to $Re^{180}$ mainly on the basis of $\beta$-decay systematics. While studying $Ta^{181}(\alpha,xn)$ excitation functions in the bombarding energy range 40–65 MeV, Daly *et al.*[8] were unable to detect the annihilation radiation of 20-h $Re^{180}$ even though the cross section of the reaction $Ta^{181}(\alpha,5n)Re^{180}$ was expected to go through a maximum of more than 1 b at about 56-MeV bombarding energy. We have therefore reinvestigated the decay of $Re^{180}$.

TABLE II. Principal $\gamma$ rays observed in the decay of $Re^{179}$.

| Energy (keV) | Relative intensity[a] |
|---|---|
| *K* x ray | 880 |
| 82.2 | 8 |
| 119.8 | 23 |
| 169.0 | 11 |
| 189.5 | 28 |
| 241$\pm$1 | 8 |
| 290.0 | 100 |
| 296.4 | 24 |
| 308.7 | 17 |
| 401.1 | 20 |
| 414.6 | 35 |
| 429.3 | 99 |
| 465$\pm$2 | 10 |
| 477.1 | 28 |
| 498.2 | 13 |

[a] Normalized to an intensity of 100 for the 290.0-keV $\gamma$ ray.

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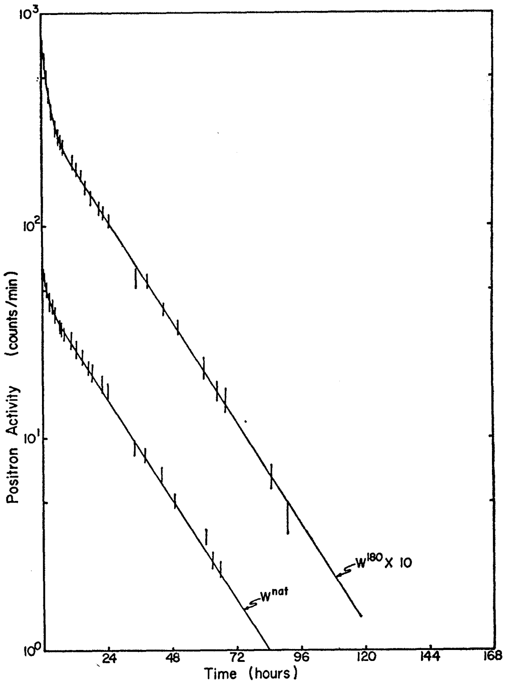

FIG. 2. Decay of the position activities induced in enriched $W^{180}$ and natural tungsten targets in bombardments with 10.2-MeV protons.

Targets of enriched $W^{180}$ (7%) and of natural tungsten were irradiated with 10.2-MeV protons. Counting of the induced activities, without chemical separations, was begun about 3 h after bombardment and was continued for several weeks. Positron intensities were measured by coincidence detection of the annihilation quanta and $\gamma$ spectra were recorded using a 3-in.×3-in. NaI(Tl) crystal. As the isotopic abundance of $W^{184}$ in each sample was accurately known, the ratio of the proton fluxes incident on the enriched and natural tungsten targets was established by measuring the relative intensity of $Re^{184}$ 0.90-MeV $\gamma$ rays in the samples about 20-days after the bombardment.

When a small amount of $F^{18}$ activity initially present had decayed away, the positron activity in each sample decayed to background with a 13–14 h half-life and there was no evidence for a longer lived $\beta^+$ component (Fig. 2). As shown in Table III the positron intensity was directly proportional to the isotopic abundance of $W^{182}$ in each target and not to the $W^{180}$ abundance. The composite 1.1–1.2-MeV $\gamma$-ray peak which was prominent in the $\gamma$-ray spectra is a well established common feature in the decay of 13-h $Re^{182}$ (spin 2) and 64-h $Re^{182}$(spin 6 or 7). In the present experiment the 64-h $Re^{182}$–13-h $Re^{182}$ isomer-production ratio was determined by decay analysis to be less than 0.065, a value which reflects the much greater probability of forming the lower-spin isomer in bombardments of zero-spin $W^{182}$ with 10-MeV protons. When the small contribution of 64-h $Re^{182}$ was subtracted, the decay rates of the 1.1–1.2-MeV photopeak and of the positron activity were identical within experimental error. It was therefore clear that the observed positron activities

TABLE III. The isotopic composition of the tungsten samples irradiated with 10.2-MeV protons and the intensities of the positron and 1.1–1.2-MeV $\gamma$-ray activities induced.

| Target | Percentage isotope abundance $W^{180}$ | $W^{182}$ | $W^{183}$ | $W^{184}$ | $W^{186}$ | $\beta^+$ intensity (arbitrary units) | 1.1–1.2 MeV $\gamma$-ray intensity (arbitrary units) |
|---|---|---|---|---|---|---|---|
| Natural W | 0.14 | 26.4 | 14.4 | 30.6 | 28.4 | 1.00 | 1.00 |
| Enriched $W^{180}$ | 6.93 | 43.7 | 11.8 | 18.8 | 1.67 | 1.67 | 1.67 |

TABLE IV. Principal $\gamma$ rays observed in the decay of 2.45-min $Re^{180}$.

| $E_\gamma$ (keV) | Relative intensity[a] |
|---|---|
| $K$ x ray | 103 |
| 103.6 | 27 |
| 511.0(annihilation radiation) | 14 |
| 827±1 | 11 |
| 902.2 | 100 |

[a] Normalized to an intensity of 100 for the 902.2-keV $\gamma$ ray.

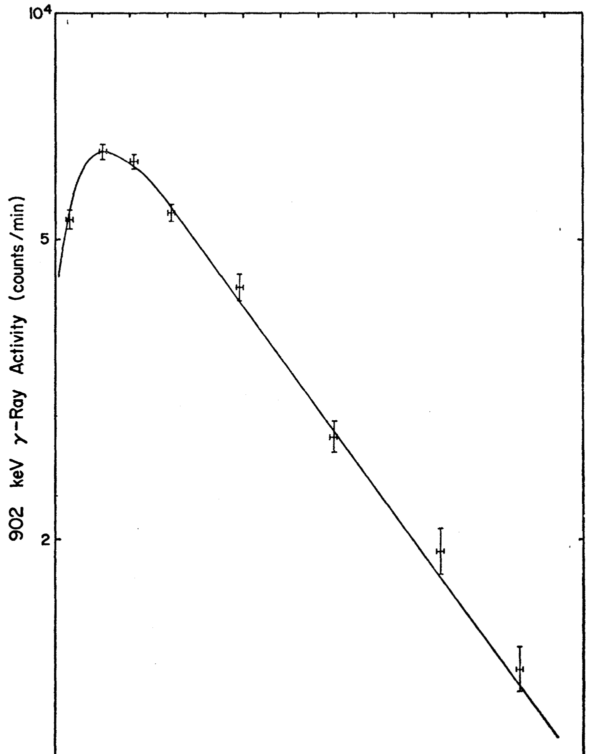

FIG. 3. Growth and decay of the 902.2-keV $\gamma$-ray activity in an osmium sample chemically separated at time, $t=0$. The full line is a calculated curve representing the production of a 2.45-min daughter activity in the decay of an initially pure 22-min parent isotope.

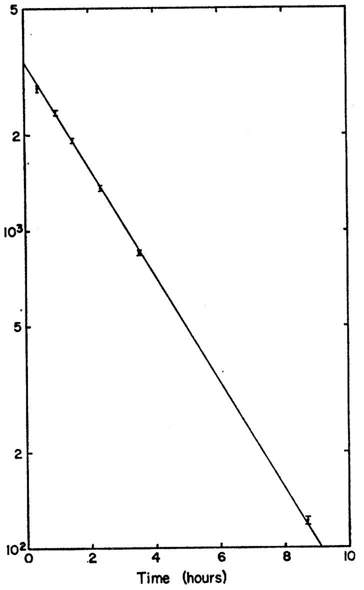

FIG. 4. The decay of the 238.6-keV $\gamma$-ray activity of $Os^{181}$.

arose in the decay of 13-h $Re^{182}$ and not of $Re^{180}$. To confirm this assignment, a tantalum foil was bombarded with $He^4$ ions of 37 MeV, the optimum bombarding energy for $Re^{182}$ production,[8] and rhenium was chemically separated. The $\beta^+$ decay rate and the intensity ratio of positrons to 13-h $Re^{182}$ $\gamma$ rays were identical with the values observed in the proton bombardments.

By assuming similar excitation functions for the reactions $W^{180}(p,n)Re^{180}$ and $W^{182}(p,n)Re^{182}$, an upper limit of less than $10^{-4}$ 20-h positrons per decay of $Re^{180}$ was calculated. The 13-h $Re^{182}$ decay scheme proposed by Harmatz *et al.*[9] was used to calculate the intensity per disintegration of all $\gamma$ rays contributing to the 1.1–1.2-MeV composite $\gamma$-ray peak and thus a branching ratio of $(3.55\pm0.40)\times10^{-3}$ positrons per decay of 13-h $Re^{182}$ was derived. This agrees within experimental error with the 13-h $Re^{182}$ $\beta^+$ branching ratio recently reported by Badalov *et al.*[10]

TABLE V. Principal $\gamma$ rays observed in the decay of 105-min $Os^{181}$.

| $E_\gamma$ (keV) | Relative intensity[a] |
|---|---|
| *K* x ray | 144 |
| 117.6 | 28 |
| 167.5 | 5 |
| 238.6 | 100 |
| 1061±1 | 10 |

[a] Normalized to an intensity of 100 for the 238.6-keV $\gamma$ ray.

It is noteworthy that the mass of $Re^{180}$ quoted in many reference books is based[11] on the existence of a 1.9-MeV $Re^{180}$ positron; it should now be deleted.

### C. The Decay of 2.45 min $Re^{180}$

Fischer[12] reported a 2.4-min $Re^{180}$ isomer decaying principally by electron capture with the emission of 106- and 880-keV $\gamma$ rays. We have produced this isotope in 32-MeV $He^3$ ion and 65-MeV $He^4$ ion bombardments of tantalum and have confirmed that its half-life is 2.45±0.10 min; we have also confirmed the mass assignment in 10-MeV proton bombardments of enriched $W^{180}$ and natural tungsten targets. As no other activity which could be assigned to $Re^{180}$ was observed in any of these bombardments, it can be assumed that the 2.45-min activity corresponds to the decay of the $Re^{180}$ ground state. The most intense $\gamma$ rays observed are listed in Table IV. The 103.6-keV $\gamma$ ray obviously corresponds to the well known $2^+-0^+$ transition in the ground-state rotational band of $W^{180}$; however, $\gamma$ rays corresponding to the $6^+-4^+$ and $4^+-2^+$ transitions in this band were not observed in the spectra. The intense 902.2-keV $\gamma$ ray corresponds closely in energy with a strong 904-keV transition observed by Graetzer *et al.*[13] in conversion electron spectroscopy of the levels of $W^{180}$ populated in the reaction $Ta^{181}(p,2n)W^{180}$. The decay of $Re^{180}$ will be discussed fully in Ref. 1, when the $\gamma$-$\gamma$ coincidence results will be presented.

### D. The Decay of $Os^{180}$

Owing to severe difficulties encountered in obtaining good coincidence data for 2.45-min $Re^{180}$ because of its short half-life, a search was made for its even-even precursor $Os^{180}$, an isotope which had not previously been identified. Enriched $W^{180}$ (7%) was bombarded with 32-MeV $He^3$ ions and fast osmium chemistry was performed. The 103.6- and 902.2-keV $\gamma$ rays characteristic of $Re^{180}$ decay were observed to grow in before decaying finally with a half-life of about 22 min. In Fig. 3, the growth and decay of the 902.2-keV $\gamma$-ray activity is seen to follow the behavior expected for a 2.45-min daughter activity growing in and reaching transient equilibrium with an initially pure 22-min parent isotope. Two repetitions of this experiment gave confirmatory results. The 2-min osmium activity could not be detected in enriched $W^{182}$ (92%) targets bombarded with 32-MeV $He^3$ ions, although $Os^{181}$ and other osmium activities were produced in high intensity; on the other hand, bombardment of enriched $W^{182}$ with 68-MeV $\alpha$ particles produced intense sources of the 22-min activity. The evidence favoring the assignment of the new activity to $Os^{180}$ is therefore rather

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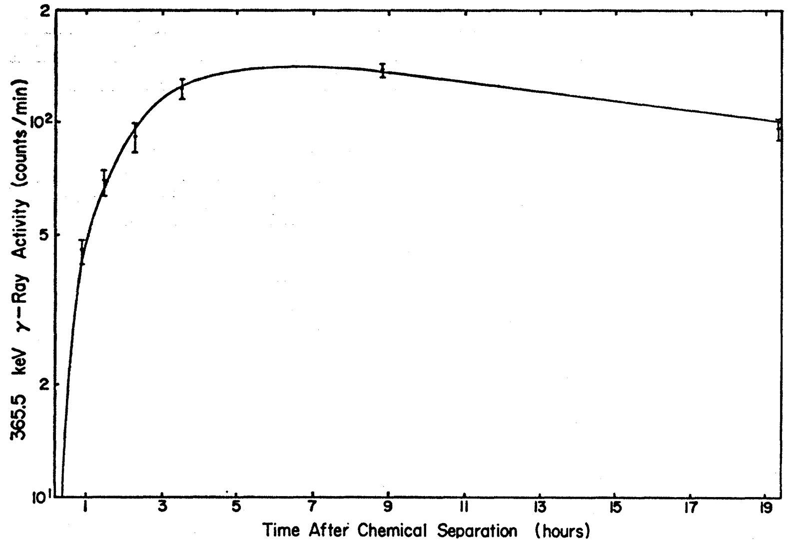

FIG. 5. Growth and decay of the 365.5-keV $\gamma$-ray activity of $Re^{181}$ in an osmium sample chemically separated at time, $t=0$. The full line is a calculated curve representing the production of a 20-h daughter activity in the decay of an initially pure 105-min parent isotope.

conclusive. Using the intense sources produced in the 68-MeV $\alpha$-particle bombardments, the half-life of $Os^{180}$ was determined to be 21.7±0.6 min. It decays by electron capture but does not emit any strong characteristic $\gamma$ rays.

A 23-min osmium isotope emitting 93- and 101-keV conversion electrons and produced in high-energy proton bombardments of $Re^{185}$, was assigned by Foster *et al.*[2] to $Os^{181}$. We have found (see below) that this assignment is not correct. It is very probable that Foster *et al.* observed the decay of $Os^{180}$, and that the 93- and 101-keV conversion lines corresponded to *L* and *M* conversion, respectively, of the 103.6-keV transition in the decay of $Re^{180}$.

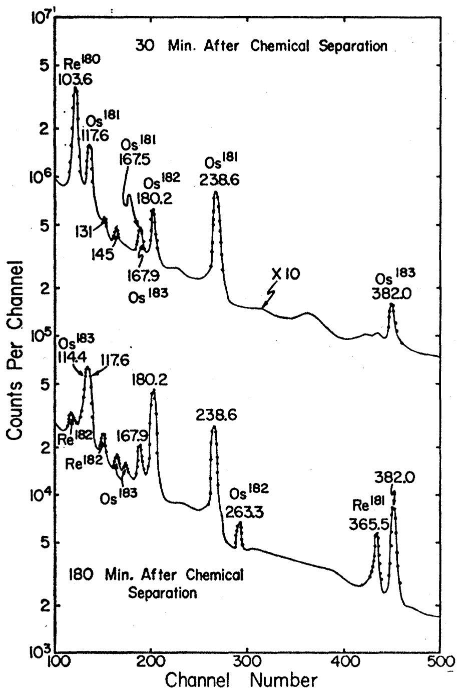

FIG. 6. $\gamma$-ray spectra taken 30 min and 180 min after chemical separation of osmium from an enriched $W^{182}$ target which had been bombarded with 68-MeV $\alpha$ particles.

### E. The Decay of $Os^{181}$

Surkov *et al.*[14] have assigned to $Os^{181}$ a 2.7-h osmium activity produced in 660-MeV proton bombardments of gold. An intense 230-keV $\gamma$ ray characterized the decay, and the mass assignment was confirmed by $Re^{181}$ milking experiments. We have produced $Os^{181}$ in 32-MeV $He^3$ ion and 65-MeV $He^4$ ion bombardments of enriched $W^{182}$. This isotope decays by electron capture with a half-life of 105±3 min (Fig. 4) and the energies; and relative intensities of its principal $\gamma$ rays are listed in Table V. Diamond *et al.*[15] have reported an osmium isotope which emits 117-, 236- and 510-keV $\gamma$ rays and decays with a half-life of about 100 min; they tentatively assigned it to $Os^{183m}$. However, we have carefully measured the growth of the 365.5-keV $\gamma$ ray of $Re^{181}$ into an initially pure osmium sample, and have found that the parent of $Re^{181}$ has a half-life of 105±15 min (Fig. 5). The high quality of the spectral data is indicated in Fig. 6, which shows clearly the marked changes produced in the low-energy $\gamma$ spectrum as the 105-min osmium activity decays into 20-h $Re^{181}$. The 105-min activity is therefore assigned to $Os^{181}$ with confidence. The 510-keV $\gamma$ ray showed no 105-min decay component and appears to belong exclusively to 22-h $Os^{182}$.

Evidence for a 2.9-min isomer of $Os^{181}$, emitting strong 117- and 142-keV $\gamma$ rays has also been observed in bombardments of enriched $W^{182}$ with 32-MeV $He^3$ ions

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but a further experiment is necessary to establish the mass assignment firmly.

## ACKNOWLEDGMENTS

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# Test of Statistical Theory of Nuclear Reactions at 24 keV

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Neutron-activation cross sections have been measured for about 40 nuclei. An Sb-Be photoneutron source was used for the measurements. The cross sections were measured relative to $I^{127}$, whose cross section was taken equal to 0.82 b. With these measured values of the cross sections, calculations were made using the statistical theory of nuclear reactions, to obtain information about the parameter $\xi = D/2\pi\Gamma_\gamma$, where $D$ is the average level spacing and $\Gamma_\gamma$ the radiation width. These values of $\xi$ were compared with those obtained from low-energy resonance parameters. In general there is good agreement between the calculated and the observed values of $\xi$, which shows that the statistical theory is valid at this energy for all types of nuclei. It was also found that $\xi$ is a smoothly varying function of the capture cross section $\sigma$.

## I. INTRODUCTION

NEUTRON-ACTIVATION cross-sections at 24 keV have been measured for about 40 nuclei to complete the data on cross sections at this energy. Cross sections in the keV energy region are helpful in the design of reactors and in the study of the cosmological theory of element formation,[1] as well as in the study of nuclear reaction theories.[2–5] All cross sections were measured relative to $I^{127}$, whose cross section was taken equal to 0.82 b.[6,7] In all cases we used a thin-window beta counter for measuring the activation cross sections.

For very low energy one gets resonances in the neutron-capture cross sections which can be explained nicely by Breit-Wigner formulas.[8] Some calculations[9] of the capture cross sections have been performed in the energy range of 0.030 to 4.0 MeV for $Hf^{180}$, $Ta^{181}$, $W^{186}$, $Au^{197}$, and $Th^{232}$ nuclei. These workers have compared the calculated value of the cross section with the experimentally measured value. In doing so they obtained the value of a parameter $\xi = D/2\pi\Gamma_\gamma$ (where $\Gamma_\gamma$ is radiation width and $D$ is the average level spacing) which gives the best fit. This value of $\xi$ was compared with the value obtained from low-energy resonance parameters.[10] It was found that, except in the case of $Th^{232}$, the calculated value of $\xi$ is very close to the experimental value obtained from low-energy resonance parameters.

In the present work we have put the measured values of the cross sections into the expression for the capture cross section obtained from the statistical theory of nuclear reactions and have calculated the value of $\xi$ for 74 cases (from $A=23$ to $A=232$) at 24 keV. The value of $\xi$ so obtained corresponds to zero-energy neutrons.[2] From the values of the resonance parameters $\Gamma_\gamma$ (the radiation width) and $D$ (the average level spacing), the values of $\xi$ were obtained for the cases where these parameters are known. To check the validity of the statistical theory, values of $\xi$ calculated from the statistical theory are then compared with the values of $\xi$ obtained from resonance parameters.

## II. EXPERIMENTAL PROCEDURE

The Sb-Be photoneutron sources were obtained from the Atomic Energy Commission, Trombay, Bombay.

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