UCRL- 757

UNCLASSIFIED

UNIVERSITY OF CALIFORNIA — BERKELEY

TWO-WEEK LOAN COPY

This is a Library Circulating Copy which may be borrowed for two weeks. For a personal retention copy, call Tech. Info. Division, Ext. 5545

RADIATION LABORATORY

DISCLAIMER

This document was prepared as an account of work sponsored by the United States Government. While this document is believed to contain correct information, neither the United States Government nor any agency thereof, nor the Regents of the University of California, nor any of their employees, makes any warranty, express or implied, or assumes any legal responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by its trade name, trademark, manufacturer, or otherwise, does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof, or the Regents of the University of California. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof or the Regents of the University of California.

COPY 2

UCRL 757
Unclassified Distribution

UNIVERSITY OF CALIFORNIA

Radiation Laboratory

Contract No. W-7405-eng-48

EXCITATION FUNCTIONS FOR SPALLATION REACTIONS ON Cu

F. O. Bartell, A. C. Helmholz,

S. D. Softky, and D. B. Stewart

June 20, 1950

Berkeley, California

INSTALLATION	No. of Copies
Argonne National Laboratory	8
Armed Forces Special Weapons Project	1
Atomic Energy Commission, Washington	2
Battelle Memorial Institute	1
Brush Beryllium Company	1
Brookhaven National Laboratory	8
Bureau of Medicine and Surgery	1
Bureau of Ships	1
Carbide and Carbon Chemicals Div., Union Carbide and Carbon Corp. (K-25 Plant)	4
Carbide and Carbon Chemicals Div., Union Carbide and Carbon Corp. (Y-12 Plant)	4
Chicago Operations Office	1
Cleveland Area Office, AEC	1
Columbia University (J. R. Dunning)	2
Columbia University (G. Failla)	1
Dow Chemical Company	1
H. K. Ferguson Company	1
General Electric Company, Richland	3
Harshaw Chemical Corporation	1
Idaho Operations Office	1
Iowa State College	2
Kansas City Operations Branch	1
Kellex Corporation	2
Knolls Atomic Power Laboratory	4
Los Alamos Scientific Laboratory	3
Mallinckrodt Chemical Works	1
Massachusetts Institute of Technology (A. Gaudin)	1
Massachusetts Institute of Technology (A. R. Kaufmann)	1
Mound Laboratory	3
National Advisory Committee for Aeronautics	2
National Bureau of Standards	2
Naval Radiological Defense Laboratory	2
New Brunswick Laboratory	1
New York Operations Office	5
North American Aviation, Inc.	1
Oak Ridge National Laboratory	8
Patent Branch, Washington	1
Rand Corporation	1
Sandia Laboratory	1
Sante Fe Operations Office	1
Sylvania Electric Products, Inc.	1
Technical Information Division, Oak Ridge	15
USAF, Air Surgeon (R. H. Blount)	1
USAF, Director of Armament (C. I. Browne)	1
USAF, Director of Plans and Operations (R. L. Applegate)	1
USAF, Director of Research and Development (F. W. Bruner and R. J. Mason)	2
USAF, Eglin Air Force Base (A. C. Field)	1

INSTALLATION	No. of Copies
USAF, Kirtland Air Force Base (M. F. Cooper)	1
USAF, Maxwell Air Force Base (F. N. Moyers)	1
USAF, NEPA Office	2
USAF, Office of Atomic Energy (A. A. Fickel and H. C. Donnelly)	2
USAF, Offutt Air Force Base (H. R. Sullivan, Jr.)	1
USAF, Wright-Patterson Air Force Base (Rodney Nudenberg)	1
U. S. Army, Atomic Energy Branch (A. W. Betts)	1
U. S. Army, Army Field Forces (James Kerr)	1
U. S. Army, Commanding General, Chemical Corps Technical Command (J. A. MacLaughlin thru Mrs. G. Benjamin)	1
U. S. Army, Chief of Ordnance (A. R. Del Campo)	1
U. S. Army, Commanding Officer Watertown Arsenal (C. H. Deitrick)	1
U. S. Army, Director of Operations Research (Ellis Johnson)	1
U. S. Army, Office of Engineers (Allen O'Leary)	1
U. S. Army, Office of the Chief Signal Officer (Curtis T. Clayton thru G. C. Hunt)	1
U. S. Army, Office of the Surgeon General (W. S. Stone)	1
U. S. Geological Survey (T. B. Nolan)	1
U. S. Public Health Service	1
University of California at Los Angeles	1
University of California Radiation Laboratory	5
University of Rochester	2
University of Washington	1
Western Reserve University	2
Westinghouse Electric Company	4
University of Rochester (R. E. Marshak)	1
California Institute of Technology (R. F. Bacher)	1
Total	144

Information Division
Radiation Laboratory
University of California
Berkeley, California

EXCITATION FUNCTIONS FOR SPALLATION REACTIONS ON Cu

F. O. Bartell, A. C. Helmholz, S. D. Softky, and D. B. Stewart

Radiation Laboratory, Department of Physics,
University of California
Berkeley, California

June 20, 1950

Abstract

Excitation functions have been measured for 190 Mev deuterons from the 184-inch Berkeley cyclotron on the natural isotopic mixture of Cu^{63} and Cu^{65}. Chemical separation of Zn, Cu, Ni, Co, Fe, and Mn has been performed and the yields of several of the radioactive isotopes of these elements have been measured as a function of deuteron energy. Results will be discussed which seem in accord with Serber's theory of high energy processes and with other information on the yields of nuclear reactions at these energies.

EXCITATION FUNCTIONS FOR SPALLATION REACTIONS ON Cu

F. O. Bartell, A. C. Helmholz, S. D. Softky, and D. B. Stewart

Radiation Laboratory, Department of Physics,
University of California
Berkeley, California

June 20, 1950

Measurements of the yields of nuclear reactions as a function of the energy the bombarding particles have provided considerable information regarding the mechanics of the nuclear processes involved. At low energies the familiar type of excitation function is one which rises from the threshold to a maximum and then falls off rapidly due to the competition from other processes. Most of these results are satisfactorily explained by the theory of the compound nucleus, formed by the addition of the bombarding particle. Early results with the very high energy particles from the 184-inch cyclotron showed a considerably different behavior in that the yield did not fall off at high energies. Serber[1] has explained these results in terms of the inelastic scattering of the impinging particle leaving in turn an excited nucleus. This excited nucleus is now the compound nucleus and decays by the usual processes. More detailed calculations have been made for the case of $C^{12}(p,pn)C^{11}$ by Heckrotte and Wolff.[2]

This work was undertaken for the purpose of making a thorough study of the different radioactivities resulting from the deuteron bombardment of a single element, copper. Lindner and Perlman[3] have discussed the factors involved in the spallation reactions from As. These factors will not be reviewed here. Excitation functions have been determined for Zn^{63}, Zn^{62}, Cu^{64}, Cu^{61}, Ni^{65}, Ni^{57},

---

(1) R. Serber, Phys. Rev. 72, 1114 (1947)

(2) W. Heckrotte and P. Wolff, Phys. Rev. 73, 264 (1948)

(3) M. Lindner and I. Perlman, Phys. Rev. 78, 499 (1950)

Co^{61}, Co^{58}, Fe^{59}, Fe^{52}, Mn^{56}, Mn^{52}, formed by 194 Mev deuterons on natural isotopic copper. The stacked foil technique, described for the 184-inch cyclotron by Thornton and Senseman,(4) was used. The foils on which the chemical separations were performed were electrolytic copper .002 in. thick, 1 in. diameter discs. These were placed in a 2 in. square stack of 1/32 in. copper absorbers so as to cover the energy ranges desired. It was not practical to perform the separations on more than 12 foils, so 10 were placed between 194 and 0 Mev energies of the deuterons, 2 beyond the range to give an estimate of the neutron background and of any effect of deuterons scattered into the side of the stack. A .001 in. Al foil of the same size as the Cu foils was placed at the high energy edge of the stack and used as a monitor. The $Al^{27}(d,\alpha p)Na^{24}$ cross section .048 barns(5) at 194 Mev was used to estimate the cross sections for the elements formed.

The stack of foils was bombarded in the internally deflected beam of the 184-inch cyclotron for times of the order of one hour. After chemical separations to be described below, the samples were counted with mica end window counters, and the resultant decay curves resolved to determine amounts of different isotopes present. No special attempt was made to determine isotopes with half-lives less than about one hour. The Al monitor was counted under, as nearly as possible, the same geometry in order to minimize effects of backscattering, etc.; that is, if the samples were plated on Pt discs, the monitor was mounted on such a disc, etc. Experiments showed variations in counting rates of as much as 100 percent, depending on the backing and geometry of the sample, so the estimates

---

(4) R. L. Thornton and R. W. Senseman, Phys. Rev. 72, 872 (1947)

(5) H. W. Hubbard, Phys. Rev. 75, 1470 (1949)

of cross section are certainly no more accurate than to a factor of 2. Fortunately, the 1.4 Mev β-spectrum of Na^{24} is a reasonable mean of those investigated, though this isotope was chosen because of the convenient half-life and absence of other activities. The excitation curve for the reaction $Al^{27}(d,\alpha p)Na^{24}$ was also used as a check on the energy values. It has a peak at approximately 25 Mev, which was the energy at the last Cu foil. The deuterons have passed through 22 g/cm^2 and the energy straggling is so large that no detail is possible below this.

Experimentally the major problem was that of getting quantitative chemical separation of the elements near Cu from the 400 mg of Cu in the foils, and using at maximum about 3 mg of carrier. More carrier would have affected β-counting accuracy. The problem was met by special methods of separation and duplicate equipment for processes such as ether extraction, etc., so that work on 12 foils could be completed in a few hours. The details are given in the appendix.

Discussion of Results[6]

Zn^{63} and Zn^{62}

Zn^{63} is the well-known 38 min. β^+ activity formed in this case by (d,2n) and (d,4n) reactions; Zn^{62}, formed by (d,3n) and (d,5n), decays 90 percent by K capture, 10 percent by β^+ emission, and is mainly detected by its daughter, the 10 min. Cu^{62}.[7] The yields are shown on Fig. 1. These activities have the advantage that there is no background due to neutrons since Zn can be formed only by the addition of a charge. Each curve is presumably a composite of two double humped ones due to the two reactions but the energy resolution is not sufficient to show this up. The Zn^{62} curve is displaced to higher energy as

(6) The periods, radiations, etc. of the radioactive isotopes are given in the table of Seaborg and Perlman, Rev. Mod. Phys. 20, 585 (1948)

(7) R. W. Hayward, Phys. Rev., in press.

would be expected. The fall off at high energies is not so rapid as might be expected from simple compound nucleus theory. This may be due to exchange collisions between high energy incident protons and neutrons in the nucleus. The cross section at the maximum, 0.4 barns, is also of the expected order of magnitude, the geometrical cross section of a Cu nucleus being $\sim$1 barn.

Cu^{64} and Cu^{61}

Cu^{64} is the familiar 12.8 hr. activity whose excitation curve by the reaction (d,p) has been studied by Livingston and Wright[8] and has a maximum below 10 Mev. Clearly this portion of the curve is not visible in Fig. 2 where the yield of Cu^{64} is plotted. (Note the different ordinates for Cu^{64} and Cu^{61}.) The maximum at $\sim$40 Mev is presumably due to the reaction (d,dn), (d,p2n), or (p,pn)(n,2n) depending on whether one considers the reaction as due to the deuteron as a whole or to one of its constituents. One might expect the maximum of these reactions to be at about twice the threshold energy (cf. Heckrotte and Wolff[2]) or about 20 Mev.

Cu^{61} (Fig. 2) is the well-known 3.4 hr. activity. It requires the expulsion of more neutrons and, consequently, the maximum in its curve is at higher energy than that for Cu^{64}. The shape suggests a combination of sharper resonance with slowly varying portion. Why this second portion should decrease above 140 Mev is, however, not at all clear. None of the curves calculated by Heckrotte and Wolff show such a decrease, even for the case of C, a much more transparent nucleus. The maximum cross section for the production of Cu^{61} is also smaller than that for Cu^{64} as would be expected from the additional competition at higher energies.

---

(8) R. S. Livingston and B. T. Wright, Phys. Rev. 58, 656 (1940)

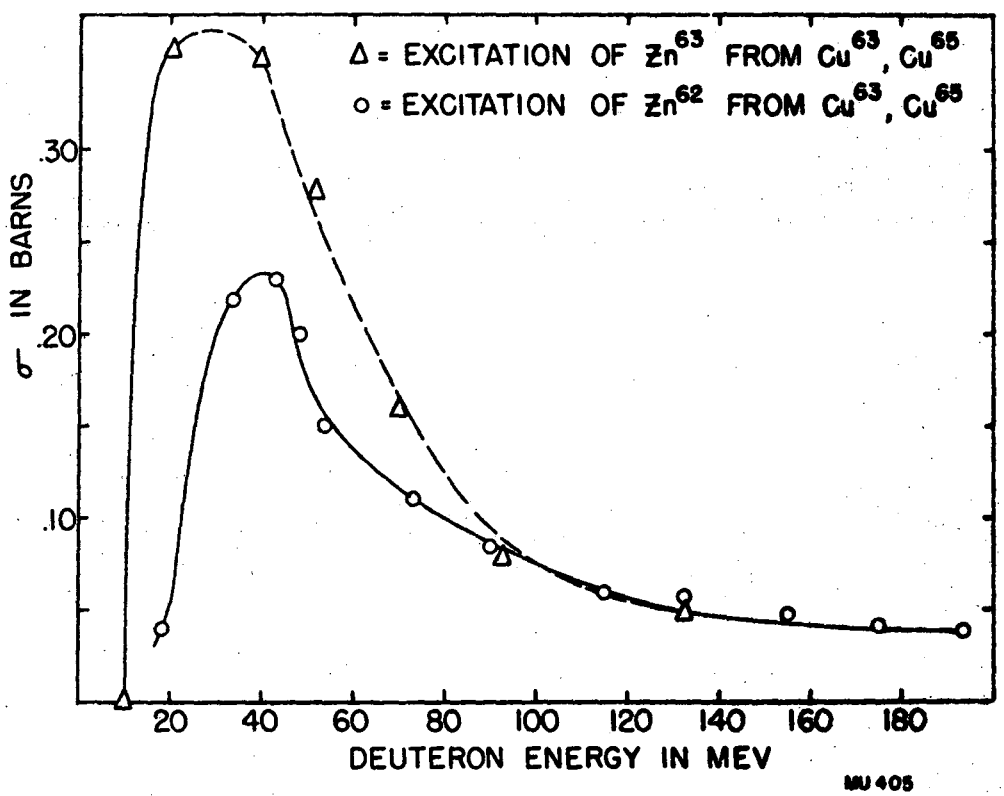
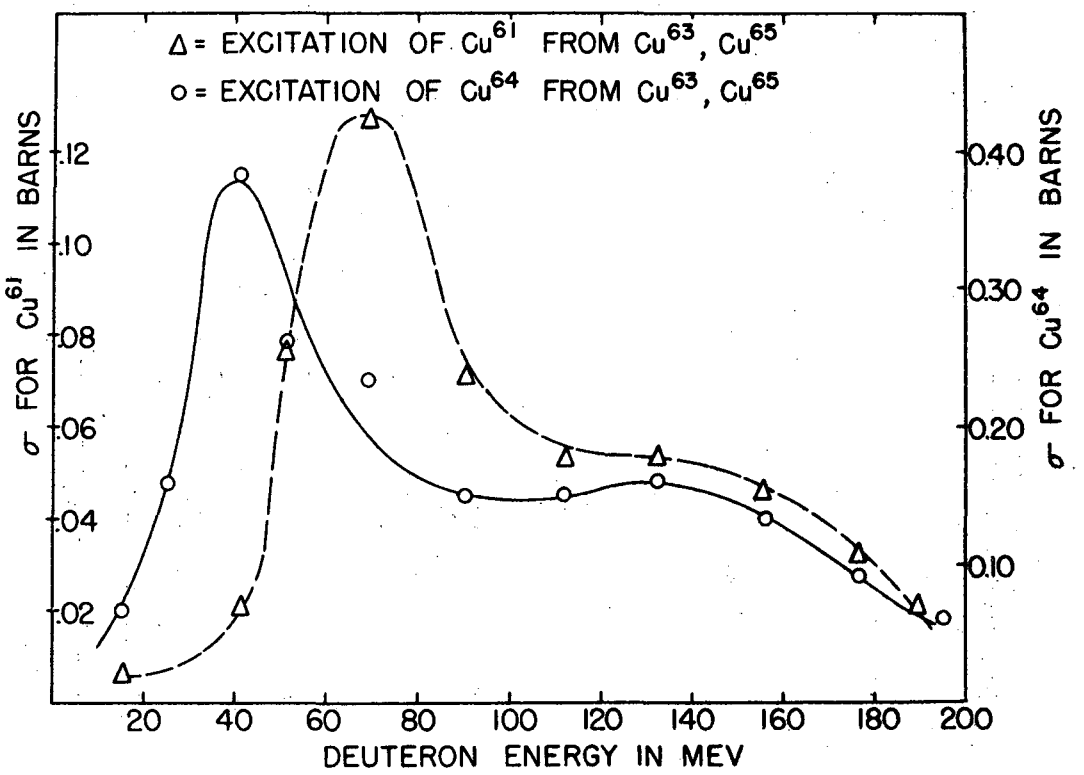

Δ = EXCITATION OF Zn63 FROM Cu63, Cu65
o = EXCITATION OF Zn62 FROM Cu63, Cu65
σ IN BARNS
.30
.20
.10
20
40
60
80
100
120
140
160
180
DEUTERON ENERGY IN MEV
MU 405

FIG. 1


Δ = EXCITATION OF Cu61 FROM Cu63, Cu65
o = EXCITATION OF Cu64 FROM Cu63, Cu65
σ FOR Cu61 IN BARNS
.12
.10
.08
.06
.04
.02
σ FOR Cu64 IN BARNS
0.40
0.30
0.20
0.10
20
40
60
80
100
120
140
160
180
200
DEUTERON ENERGY IN MEV

MU 404

FIG. 2

Ni

The two Ni isotopes measured were Ni^{65} and Ni^{57}. The circled points on Fig. 3 were adjusted to fit a smooth Ni^{65} curve. When so adjusted, the Ni^{57} points fit on a smooth curve. This seems good evidence that there was chemical loss in these samples. The magnitude of the corrections was about 20 percent. Ni^{65} is formed by the (d,2p) or (n,p) reactions, and so is present even beyond the range of the deuterons due to neutrons formed in the stack. The threshold for the reaction is low (2.3 Mev), and since the straggling is much greater than 2 Mev, a yield should be observed down to 0 Mev. The rather broad apparent maximum in the curve at about 60 Mev probably occurs at a somewhat lower energy. This energy is undoubtedly associated with the necessity of ejecting one or two protons over the Coulomb barrier. If the stripping process in which the neutron enters the nucleus and the proton goes on, gives a large contribution, the necessary deuteron energy will be high because the neutron averages only half of it. In this connection, the yield of the (n,p) reaction as a function of neutron energy would be valuable to know.

The Ni^{57} curve is characteristic of those with high threshold energies. If one calculates the threshold for $Cu^{63}(d,2p6n)Ni^{57}$ from a semiempirical mass formula,[9] one gets 62 Mev. The agreement with the curve can be considered satisfactory. As will be seen below, there is evidence that α-particles are ejected as units, but in this case, unless the deuteron is captured to form a Zn compound nucleus, α ejections cannot give Ni^{57}.

It is probably significant that the decrease of the Ni^{65}, Zn^{62}, and Cu^{61} cross sections set in at about 60 Mev, when the Ni^{57} and others listed below start to rise.

---

(9) Cf. Orear, Rosenfeld, Schluter, "Nuclear Physics, Lectures by E. Fermi," p. 6.

Co

The chemical recoveries on Co were the least satisfactory of all those performed. The activities recovered were those of Co^{58} (72 days), Co^{56} (72 days), and Co^{55} (18 hours). In order to separate the first two, which have the same half-life, the particles were counted in a rough 180° magnetic spectrograph. The Co^{56} with a 1.5 Mev beta spectrum could then be distinguished from the 0.47 Mev spectrum of Co^{58}. A pure standard of the latter activity was also prepared by bombarding Mn with low energy alphas. Since Co^{58} decays only 15 percent by positron emission, there is a rather large correction to the observed counting rate but the measurement in the magnetic spectrograph made certain that none of the x-rays were counted.

The excitation curves are of the type which rise to a fairly constant yield between 100 and 200 Mev. The surprising thing about these curves, however, is the large yield, 0.2 barns in the case of Co^{58}; 0.04 barns in the case of Co^{56} and Co^{55}. The results of Good, Peaslee, and Deutsch[10] on the positron K capture branching should be accurate, so that there is no reason to suspect an error in this measurement of the large cross section of Co^{58}. It is to be noted that the reactions yielding Co^{58} are ones which might be written (d,dαn) and (d,dα3n). The unusually high cross section for those reactions in which alpha particles can be emitted after the excitation of the bombarded nucleus is discussed below.

Co^{57} (270 d.) is undoubtedly formed also in these experiments. Since these activities were weak, it was not possible to detect Co^{57} with certainty. However, the yields of Co^{56} and Co^{58} may be somewhat too high.

Fe

The 7.8 hr. Fe^{52}, which is the parent of the 21 m. Mn^{52m}, and the 49 d.

---

(10) Good, Peaslee, and Deutsch, Phys. Rev. 69, 313 (1946)

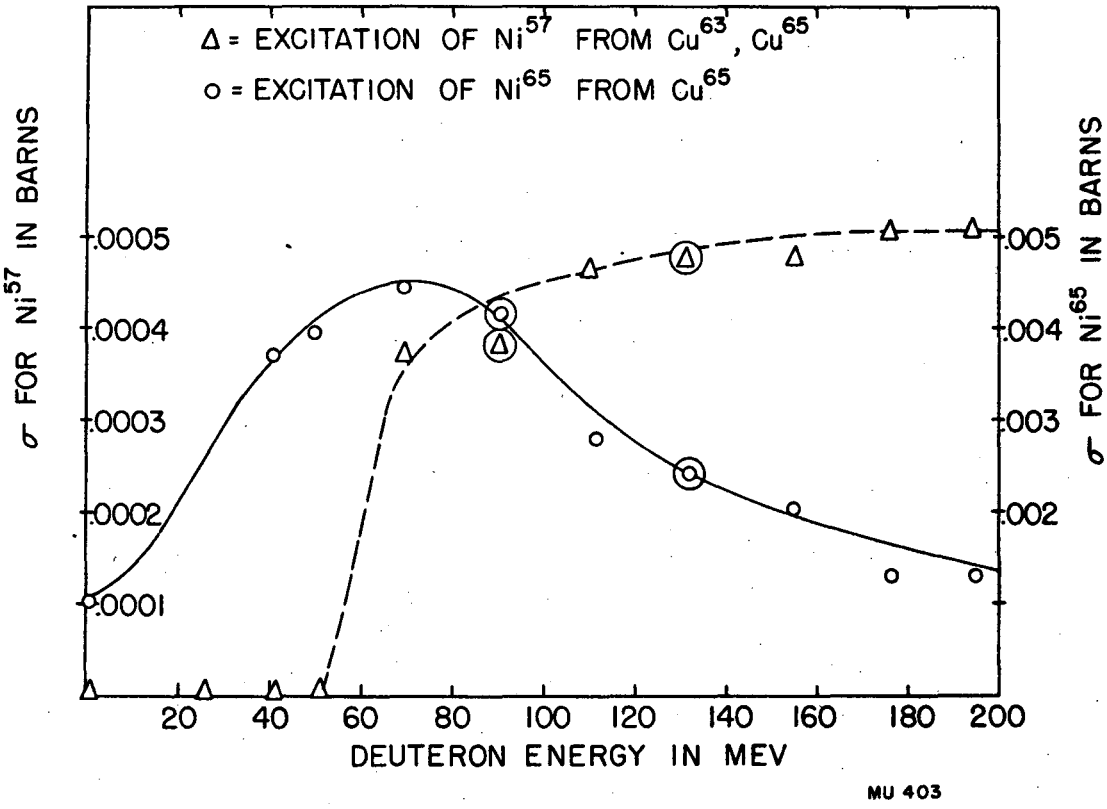

Δ = EXCITATION OF Ni57 FROM Cu63, Cu65
o = EXCITATION OF Ni65 FROM Cu65
σ FOR Ni57 IN BARNS
σ FOR Ni65 IN BARNS
.0005
.0004
.0003
.0002
.0001
.005
.004
.003
.002
20
40
60
80
100
120
140
160
180
200
DEUTERON ENERGY IN MEV
MU 403

FIG. 3

Fe^{59} were the iron activities investigated. The results are shown in Fig. 4. Corrections similar to those mentioned for Ni were made on the circled points of the Fe^{59} curve with the resultant good fit to the Fe^{52} curve.

The Fe^{59} yield extends apparently to zero energy, and while the energy definition is poor in this region, the curve does indicate that the reaction proceeds at low energies. Using the semiempirical mass formula, one finds that the reaction $Cu^{65}(d,2\alpha)Fe^{59}$ is exoenergetic by about 2 Mev. At low energies the spread in deuteron beam energy is large compared to the α barrier height, so one expects to find a yield at 0 energy. The threshold for $Cu^{63}(d,2p\alpha)Fe^{59}$ is 8 Mev, and because of the barrier for the α-particle, this reaction will not become appreciable until higher energies. The rise in the curve beginning at $\sim$ 50 Mev might be associated with this or with reactions such as $Cu^{63}(n,p\alpha)$ or $Cu^{63}(n,3p2n)$. In the latter, the threshold is 28 Mev higher, while in both, the necessary deuteron energy is increased because the neutron is considered as the particle involved and it averages only one-half the deuteron energy. Further work on these reactions using low energy deuterons is projected.

The Fe^{52} exhibits the lowest cross section and the highest threshold measured in this work. The calculated threshold for $Cu^{63}(d,4p9n)Fe^{52}$ is 102 Mev. The energetic threshold seems deinitely below this (the energy definition in this region should be good) and would indicate that a reaction such as $Cu^{63}(d,\alpha 2p7n)$ or $Cu^{63}(n,\alpha p7n)$ was participating. Such reactions are particularly necessary if one postulates that only the neutron or proton give energy to the nucleus, though it is important to remember that this cross section is very small, and occasionally a proton or neutron may impart a large fraction of the deuteron energy to a nucleus.[11]

---

(11) R. Serber, Phys. Rev. 72, 1000 (1947)

Mn

Mn^{56} (2.56 hr.) and Mn^{52} (6.5 d.) were the two Mn isotopes investigated. The curves are shown in Fig. 5. The 6.5 d. activity is the lower of the two Mn^{52} isomeric states, and is undoubtedly produced to some extent as a decay product of Fe^{52} through Mn^{52m}. The separations of Fe and Mn were performed on all samples at the same time and within a few hours of the end of the bombardment, and since the excitation curves are not radically different, the curve shown in Fig. 5 should not be much in error. Both Mn curves show the rather steep rise beginning at high energies characteristic of a reaction with high threshold. The calculated threshold for protons and neutrons out (d,5p8n) for Mn^{56} is 62 Mev, which is slightly above the observed, indicating that probably α's are involved, while the calculated threshold for Mn^{52} from Cu^{63} is 99 Mev, and the observed threshold is definitely lower. In this case it seems certain that α's rather than individual protons and neutrons are emitted.

Discussion

The sets of yield curves presented above definitely show that at high energies of the bombarding particle the typical curve predicted by the compound nucleus theory at low energies is not found. The curves are in agreement with what would be expected from the theory presented by Serber. Detailed calculations such as extensions of those of Horning and Baumhoff[12] might be worth while to show whether the agreement is as good as could be expected.

More striking is the alternation of the magnitude of cross sections with the Z of the product isotope superimposed on a decreasing trend. Table I exemplifies this at 190 Mev. Since one cannot measure the yield of every isotope, the data is only fragmentary, but it does seem that the yield of Cu is large,

(12) W. Horning and L. Baumhoff, Phys. Rev. 75, 370 (1949)

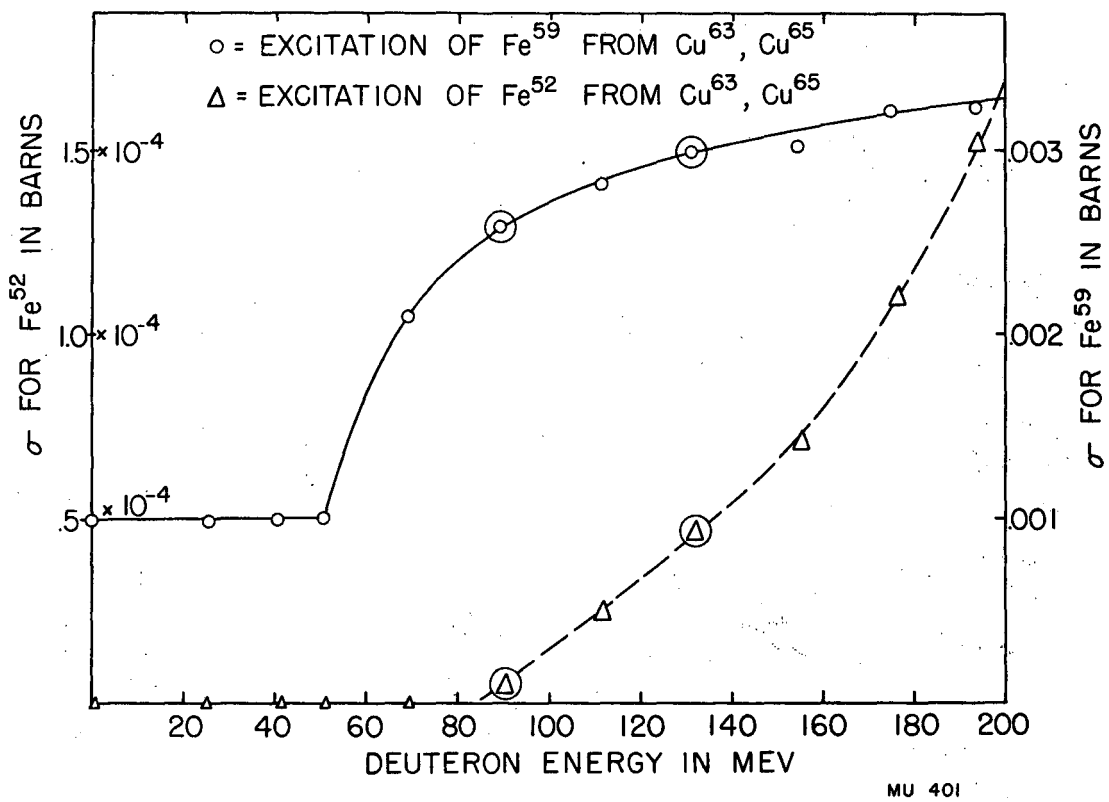

○ = EXCITATION OF Fe59 FROM Cu63, Cu65
Δ = EXCITATION OF Fe52 FROM Cu63, Cu65
σ FOR Fe52 IN BARNS
1.5 × 10-4
1.0 × 10-4
.5 × 10-4
σ FOR Fe59 IN BARNS
.003
.002
.001
20
40
60
80
100
120
140
160
180
200
DEUTERON ENERGY IN MEV
MU 401

FIG. 4

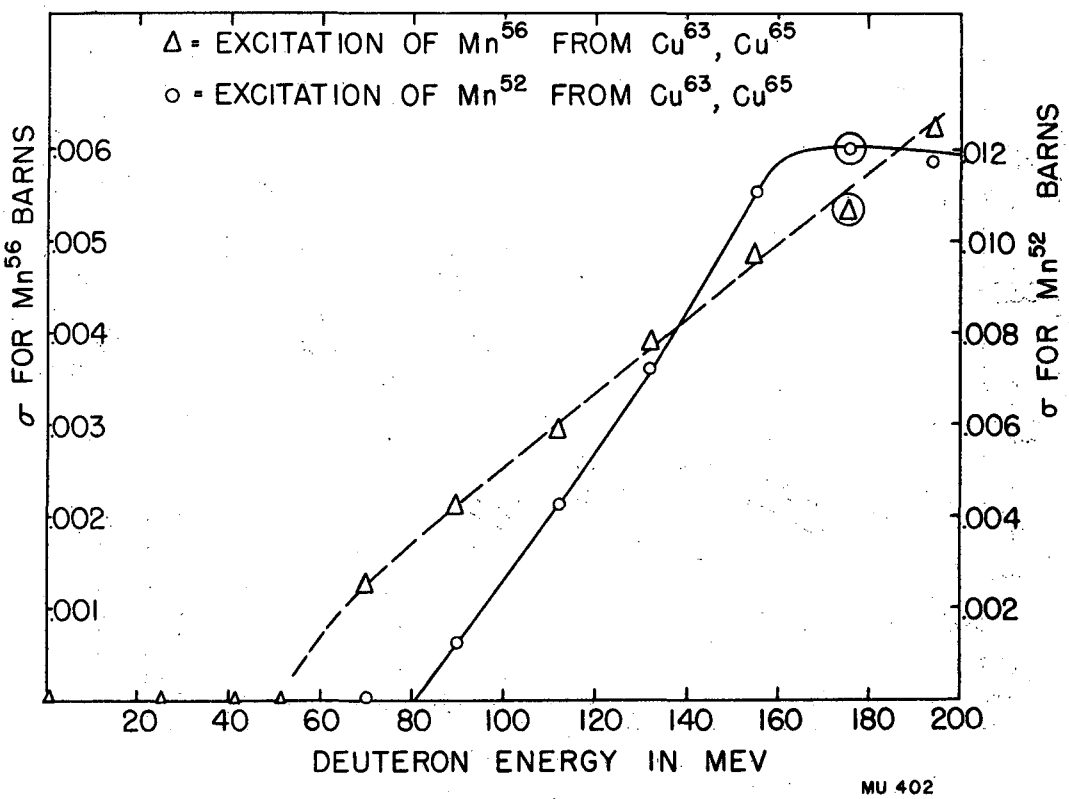

Δ = EXCITATION OF Mn56 FROM Cu63, Cu65
o = EXCITATION OF Mn52 FROM Cu63, Cu65
σ FOR Mn56 BARNS
.006
.005
.004
.003
.002
.001
σ FOR Mn52 BARNS
.012
.010
.008
.006
.004
.002
20
40
60
80
100
120
140
160
180
200
DEUTERON ENERGY IN MEV
MU 402

FIG. 5

TABLE I

Z→ / A↓	Mn	Fe	Co	Ni	Cu
61					0.11
60					
59		0.003			
58			0.200		
57				0.0005	
56	0.006		0.040		
55			0.040		
52	.012	0.00015			

MU 400

of Ni small, of Co large, etc. A good example seems to be the cases of Fe^{52} and Mn^{52}, in which the latter cross section is a factor of 100 higher although it requires the ejection of an extra proton rather than a neutron. At very high energies one might expect that any barrier effect would be negligible and that the yields would be about equal. If we assume that the incident particle neutron, proton, or deuteron, does not stick but only leaves the struck nucleus excited, then we notice that Mn^{52} can be formed by the ejection of two alphas and three neutrons from Cu^{63}, while Fe^{52} must be formed by ejection of particles less tightly bound to each other. As mentioned above, the thresholds seem to indicate that alphas may be emitted, and this alternation in yield seems to be more evidence for the same thing. Such an alternation is evident in the results of Miller, Thompson, and Cunningham[13] with Cu and also, though not so noticeably, of Hopkins and Cunningham[14] working with As.

This work was performed under the auspices of the Atomic Energy Commission.

---

(13) Miller, Thompson, and Cunningham, Phys. Rev. 74, 347 (1948)

(14) H. H. Hopkins and B. B. Cunningham, Phys. Rev. 73, 1406 (1948)

Appendix

The following chemical separations were mostly determined by convenience and the relative amounts of the activities present. The Zn separation had to be good only for Cu removal, since relative amounts of the other activities were small. The Ni, Co, Mn, and Fe separations had to be good from all other activities. The Fe separation was the most rigorous of them all, having to be good to one part in 10^6 from all other elements.

Zinc

The separation of Zn from the elements near it was as follows: Cu target foils were dissolved in the minimum necessary 6N HNO_3 and 2 mg of Zn carrier added as the nitrate or sulfate. One ml of concentrated H_2SO_4 was added and the HNO_3 removed by evaporating to SO_3 fumes. The solution was then diluted to about 3 N H_2SO_4 and 500 mg of 20 mesh Al added. This was boiled until all the Cu was reduced and for about 5 minutes after the solution appeared water white. The solution was then cooled and filtered through a rapid filter paper, the Cu metal being washed with 0.1 N H_2SO_4. The pH was adjusted to about 2 or 3 with NaOH and dilute H_2SO_4. The solution was then saturated with H_2S to precipitate ZnS, centrifuged, and the supernatant which contained Mn, Fe, Co, and Ni decanted. The precipitate was washed with 0.01 N H_2SO_4 containing H_2S and then dissolved in 3 ml concentrated HNO_3. This solution was then evaporated to dryness on a stainless steel dish, flamed to destroy any excess sulfur and water of hydration, and was ready to be counted.

Copper

The Cu target foil was dissolved in a minimum of dilute HNO_3, made up to 250 ml in a volumetric flask and some convenient small fraction (0.8 percent to 4 percent) transferred to an electrolytic cell. Two drops of concentrated H_2SO_4 and a small amount of dilute HNO_3 were added and the solution plated to

exhaustion by about 1/2 to 1 hour plating at about 20 milliamperes out of a volume of 20 ml. Zn, Mn, Fe, Co, and Ni will not plate under these conditions. The plates were quickly removed, washed in alcohol, dried in air, and were ready to count. The Cu was about 2 mg/cm^2 thick.

Nickel

The separation of Ni from the elements in the region of copper was done as follows: The target foil was dissolved in 6N HCl and a few drops of 30 percent H_2O_2, the excess H_2O_2 then being boiled off. About one-half to one mg of Ni carrier was added as chloride, the solution neutralized with NH_4OH, then made slightly acid with HCl. NH_4HSO_3 was added to reduce Cu^{++} to Cu^{+} and excess SO_2 boiled off. CuSCN was precipitated from the warm solution with a few crystals of NH_4SCN, and allowed to settle for ten minutes. The solution was filtered and the precipitate washed with a one percent solution of NH_4SCN containing a little NH_4SO_3. To complex the Fe, one ml of 50 percent tarataric acid was added to the filtrate which was made slightly ammoniacal and brought to near boiling. The Co, Zn, and Cu were thus complexed in the NH_4OH and Ni dimethyl glyoxime was precipitated by adding one to two ml of 1 percent dimethyl-glyoxime in ethanol. The precipitate was filtered and washed with hot water, dissolved in dilute HCl, and reprecipitated twice in order to free it from occluded Zn. Finally, the clean precipitate was dissolved in concentrated HNO_3, evaporated to dryness in a stainless steel dish, flamed, and was ready to count.

Cobalt

The separation of Co from the elements near Cu presented more difficulty than most of the other separations. The method finally evolved essentially consisted of removing all contaminant elements which would plate electrolytically and then plating the remaining Co onto platinum discs out of ammoniacal solution.

The Cu target foil was dissolved in minimum amount of dilute HNO_3, 1 mg each of Co, Ni, and Mn carriers, 2 drops of concentrated H_2SO_4 were added, and the solution diluted to 20 ml. The excess Cu was removed by electrodeposition on a stainless steel strip cathode set into the beaker using about 1 to 2 amp at 2 to 3 volts. When the solution was water white, the electrodes were removed, 1 ml concentrated H_2SO_4 added and the solution evaporated to SO_3 fumes to destroy HNO_3 present. Then it was diluted to 35-50 ml and saturated with H_2S to precipitate the remaining Cu. Most of the Zn occluded on these CuS precipitates. After repetition of the procedure, the filtrate was again boiled to expel H_2S, made slightly ammoniacal and 1-2 ml of 1 percent dimethyl-glyoxime in ethanol added. The Ni and Fe precipitates were filtered off, the filtrate acidified with HNO_3 and evaporated to SO_3 fumes to destroy the alcohol, a few drops of concentrated HCl added and again taken to SO_3 fumes to insure removal of NO_3. The solution was transferred to an electrolysis cell fitted with a Pt disk cathode, made strongly ammoniacal, and Co metal deposited as a smooth, uniform, adherent plate on the cathode by plating for from 1/2 to 1 hour at about 1 to 2 amperes. MnO_2 was deposited simultaneously on the stirrer anode. Zn does not plate at these voltages. The electrolyte was then removed and replaced by distilled water with the current still on in order to prevent the Co plate from dissolving. The anode was quickly removed and the Co plate washed in alcohol and dried in air.

Manganese

It was found than Mn could be separated as part of the process for Fe isolation. The foils were dissolved in a minimum of 6N HNO_3 and 1 mg Mn carrier added as the nitrate. The solution was then diluted to about 1 N HNO_3, 0.1 g of $KBrO_3$ added, and boiled for ten minutes or until the MnO_2 was well coagulated. There was probably some occlusion of Cu, Co, and Zn on this precipitate. The

precipitate was filtered and washed with 6N HNO_3 until no Cu blue showed in the wash, then redissolved through the filter paper with concentrated HCl and 2 mg Cu carrier added. The solution was made strongly ammoniacal and a few crystals of $(NH_4)_2S_2O_8$ added. Any occluded Cu, Co, and Zn was complexed in the NH_4OH. The excess persulfate was boiled off and the MnO_2 reprecipitated and centrifuged. After washing with dilute NH_4OH, the precipitate was deposited on a stainless steel dish and dried. This separation was perhaps the least troublesome of all those done in that the time required was a minimum and the degree of purification and percentage recovery excellent.

Iron

The following separation for Fe has the advantages of high purity ($\sim 10^6$) and small amount of carrier necessary. Since the Fe yield was very small, the Fe half-lives relatively long, and the Fe^{59} β^- soft, these advantages turned out to be absolutely essential, even if recovery was not as complete as in some of the other fractions.

Cu foils were dissolved in a minimum 6N HNO_3 and diluted to 1 or 2 N HNO_3. 1 mg Fe and 1 mg Mn carrier were added as nitrates. The Mn was precipitated as MnO_2 by addition of about 0.1 g $KBrO_3$ and boiling for 10 minutes, filtered and washed. $Fe(OH)_3$ was precipitated from the filtrate by addition of excess NH_4OH and centrifuged. The Cu, Co, Ni, Zn complexed in the supernatant liquid. The $Fe(OH)_3$ was dissolved in HCl, $\sim$ 3 mg of $CuCl_2$ added, and the Fe precipitated twice more, this number of sweeps being necessary to get rid of the tremendous excess of Cu activity. The final $Fe(OH)_3$ precipitate was dissolved in three drops concentrated HCl, and transferred to a separatory funnel with 6N HCl. An equal volume of diethyl ether presaturated with 6N HCl was added and the Fe extracted. Extraction was repeated on the aqueous layer, 1 mg Fe carrier added to this aqueous layer and extracted again. All ether phases were collected with

water and the ether boiled off in a hot water bath, then $Fe(OH)_3$ precipitated from this solution. This was dissolved in several drops concentrated HNO_3, evaporated to dryness on a stainless steel dish, flamed, and was ready for counting.

Information Division
scb/6-21-50