THE UNIVERSITY OF MICHIGAN
INDUSTRY PROGRAM OF THE COLLEGE OF ENGINEERING

RADIOCHEMICAL SEPARATIONS AND ACTIVATION ANALYSIS

Progress Report 6
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Project No. 7, Contract No. AT(11-1)-70 between the University of Michigan and the U. S. Atomic Energy Commission was initiated on February 1, 1952. On November 1, 1952, Progress Report No. 1 was submitted to the Atomic Energy Commission. Progress Report No. 2 was submitted on November 1, 1953, Progress Report No. 3 was submitted on November 1, 1954, Progress Report No. 4 (AECU-3116) was submitted on November 1, 1955, and Progress Report No. 5 (AECU-3375) was submitted on November 1, 1956.

The following is a report of the work which has been completed on the project during the year of November 1, 1956 to October 31, 1957.

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I FACILITIES

A. Michigan Reactor

The Michigan research reactor finally went critical on September 19, 1957 and is at the present time undergoing calibration. It had been hoped (1) that the reactor would be operating routinely during most of the past year. A number of difficulties were encountered, however, in meeting specifications for building tightness, etc. thus delaying start-up a few months. On May 14, 1957 the reactor crew passed their safety inspection in good order but several months passed while the AEC reviewed the reactor operating procedures before the operating license was issued (on September 16).

Present plans indicate that several months will be spent on calibration of the reactor, first at very low power (a few watts) and then at increasing powers up to 100 kilowatts which is the maximum permitted under the start-up license. Included in these calibration runs will be experiments to calibrate the pneumatic tube positions under varying control rod configurations and conditions of operation. It will probably not be until the first of the year, however, before the reactor will be in routine operation servicing all departments of the campus.

The reactor is a modified "swimming pool" or "Geneva" type that has been designed for 1 megawatt operation at a flux of 10^{13} n/cm^2 sec at full power. Figure 1 shows a view of the top and interior of the reactor pool while Fig. 2

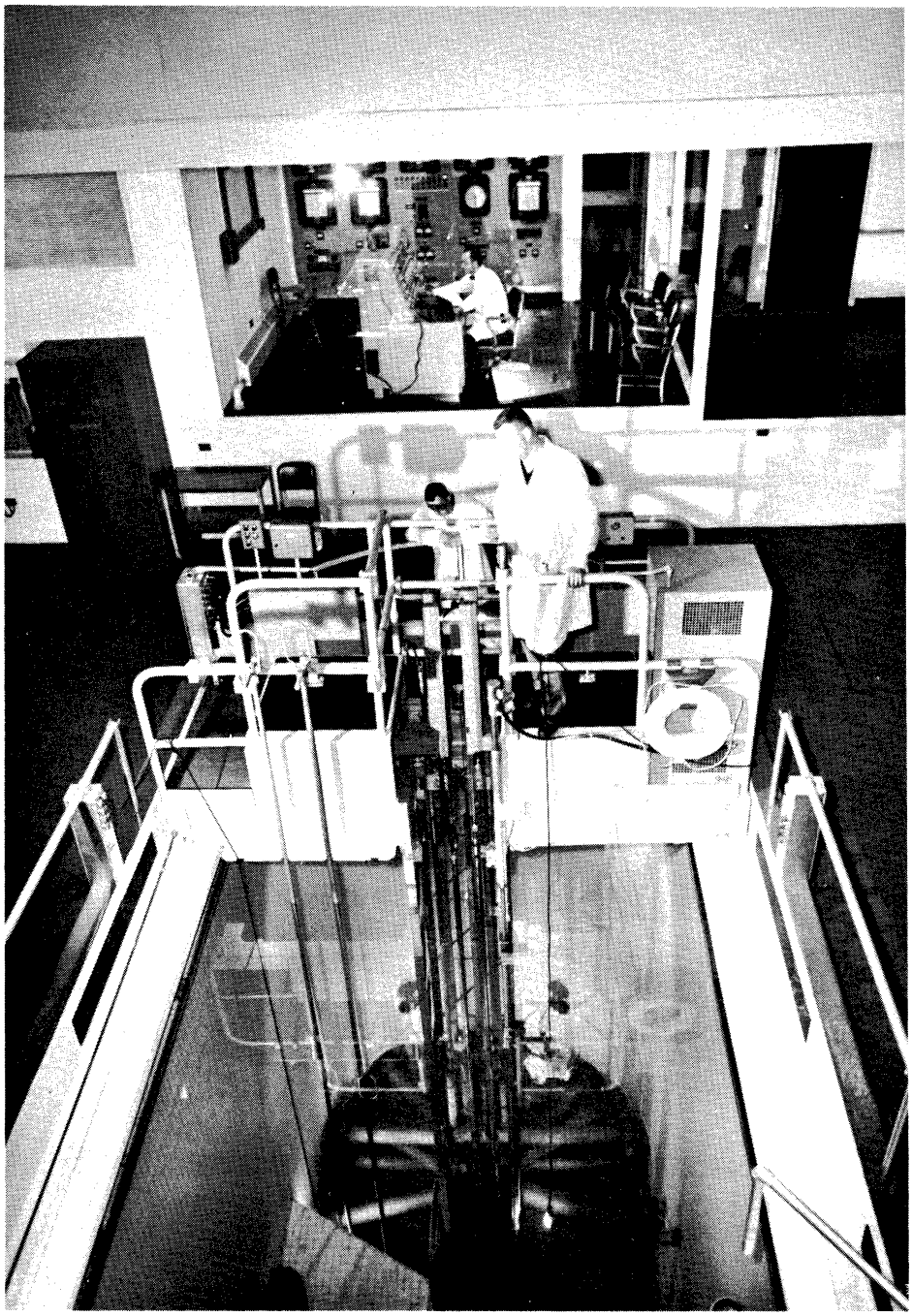


Figure 1. (view 1) Top of U. of M. Reactor Pool.

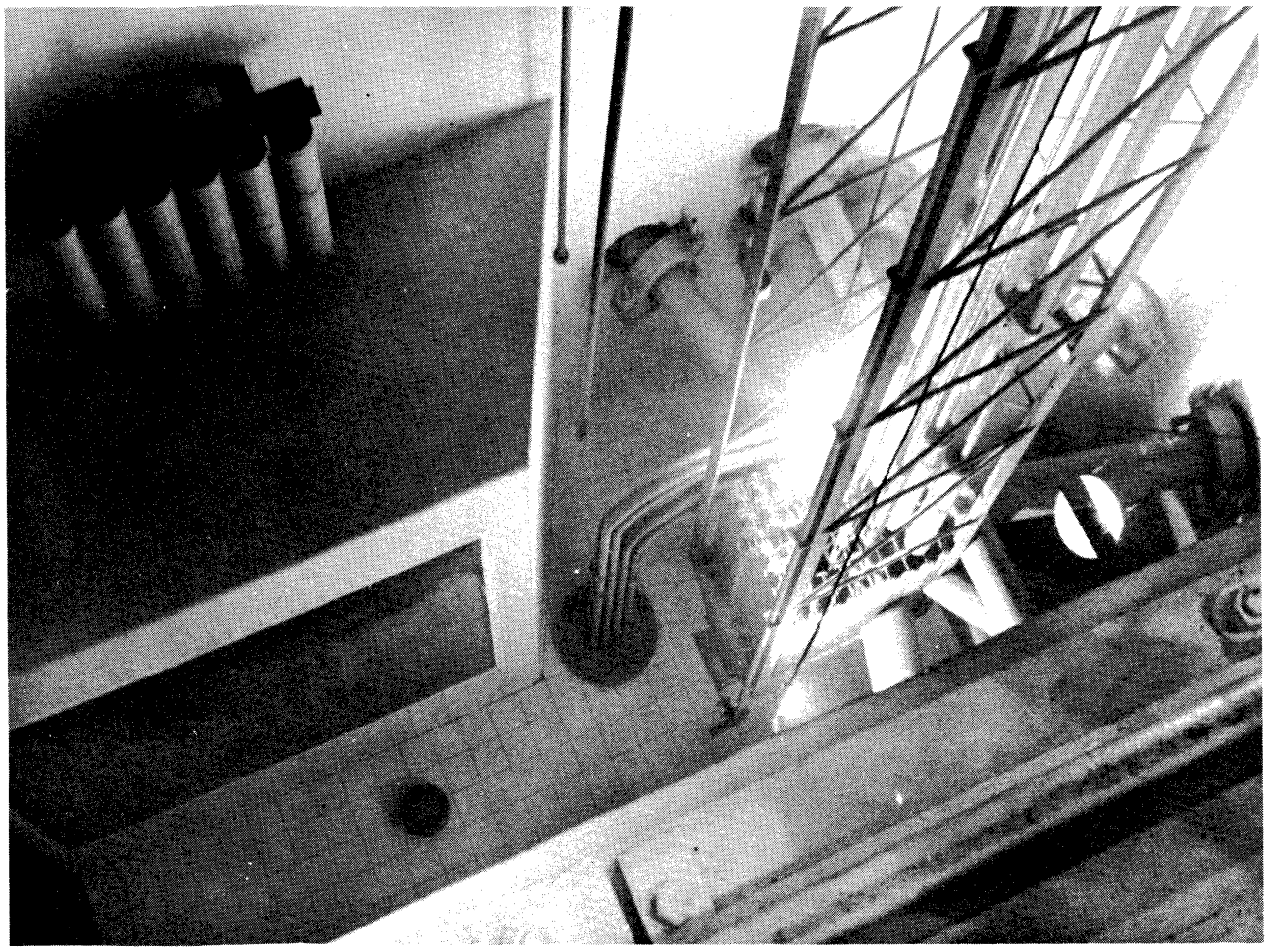


Figure 1. (view 2) Interior of U. of M. Reactor Pool.

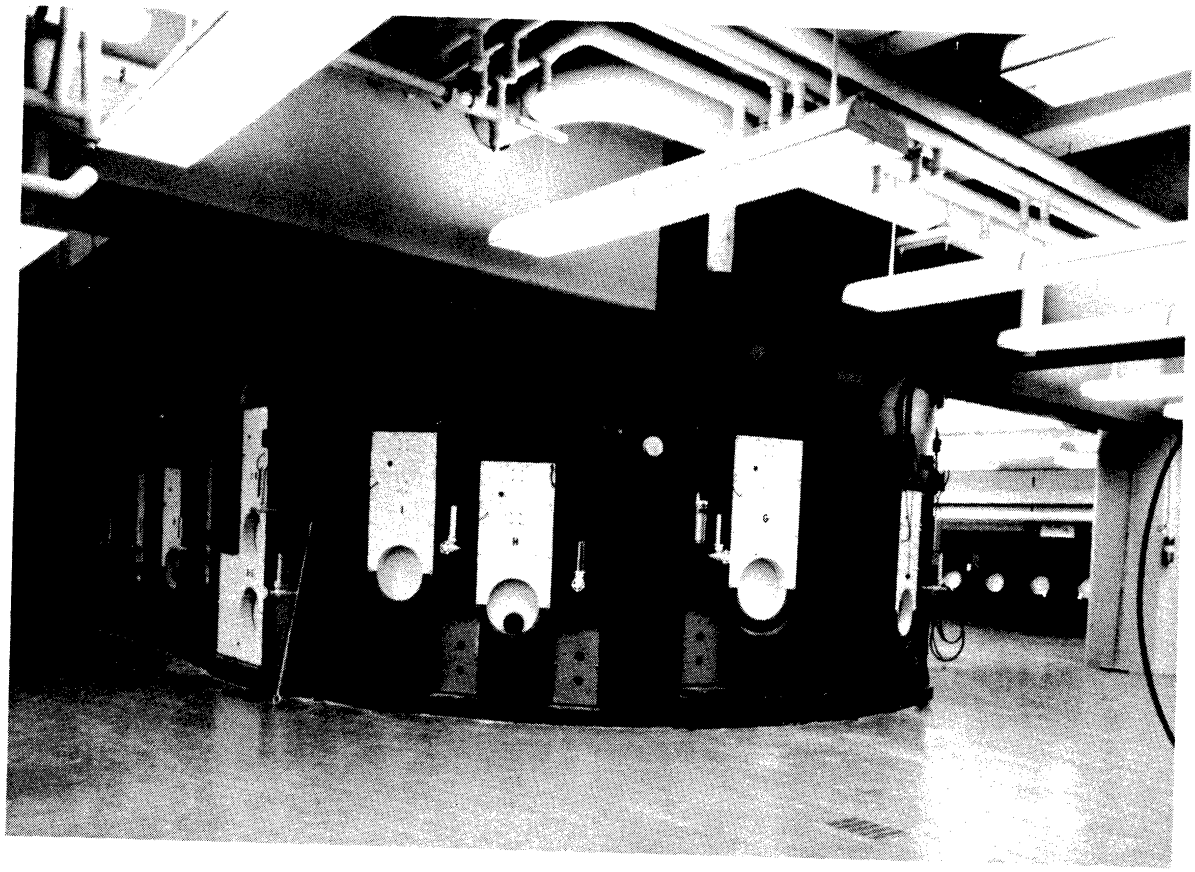


Figure 2. Beam Port Floor. (view 1)

Figure 2. Beam Port Floor. (view 2)

pictures the beam port floor. One of the pneumatic tube stations in the hoods of the Phoenix Laboratory is shown in Fig. 3, while the control timers for the pneumatic tube system are shown in Fig. 4. Rabbits of different materials can be introduced at the hood station; the tubes terminate near the edge of the core. After a predetermined irradiation time (set on the timer) the rabbit is sucked out of the reactor into the hood in a matter of five to six seconds. The system operates by vacuum rather than compressed air thus minimizing the possibility of contaminating the receiving station by radioactive dust from the reactor. The system has four pneumatic tube positions at the reactor core but there are six possible sending stations, four in the Phoenix Building and two in the reactor building. Figure 5 shows the interconnecting system in the basement of the reactor building which makes it possible to connect any one sending station with any reactor position.

B. Michigan Phoenix-Memorial Laboratory

The group has continued to make use of the hot lab facilities in this laboratory. In one building modification now being made an area about 12' x 15' is being converted from unfinished laboratory space into a specialized counting room. This room is situated next to the present counting room of the Phoenix Building and is being air conditioned (with controlled humidity) to house the 100-channel analyzer and spectrometer equipment for the activation analysis program.

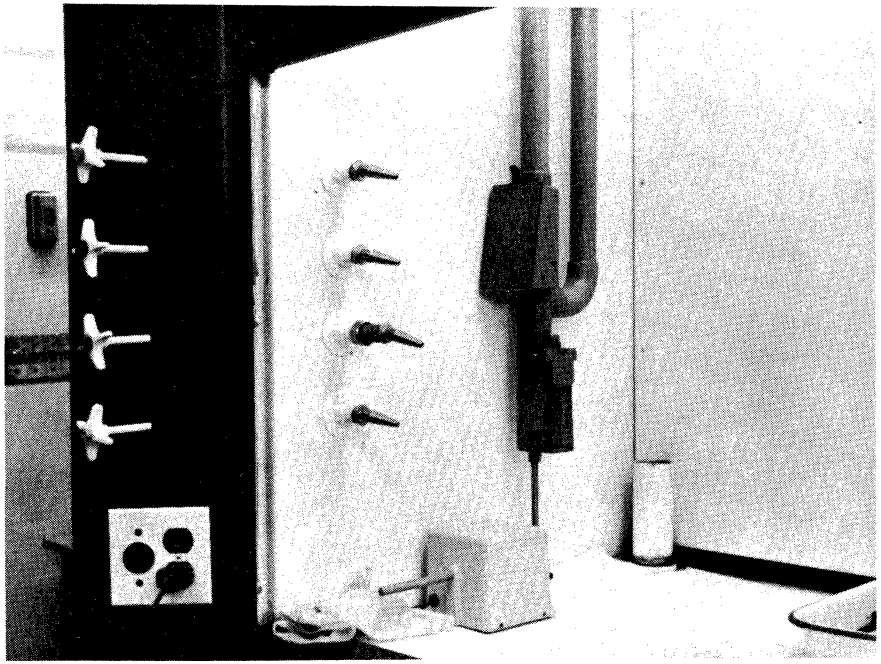
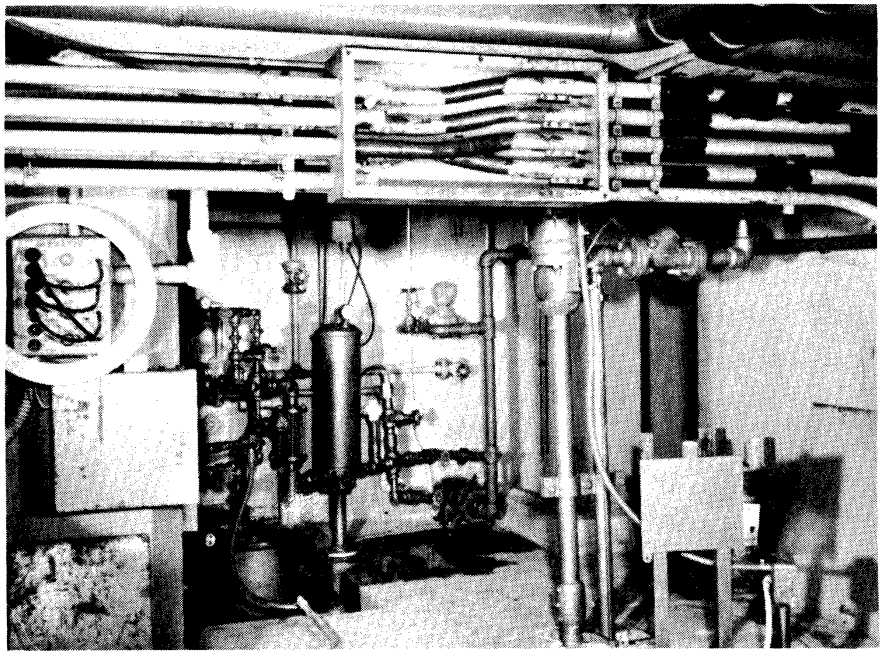


Figure 3. Rabbit Station.



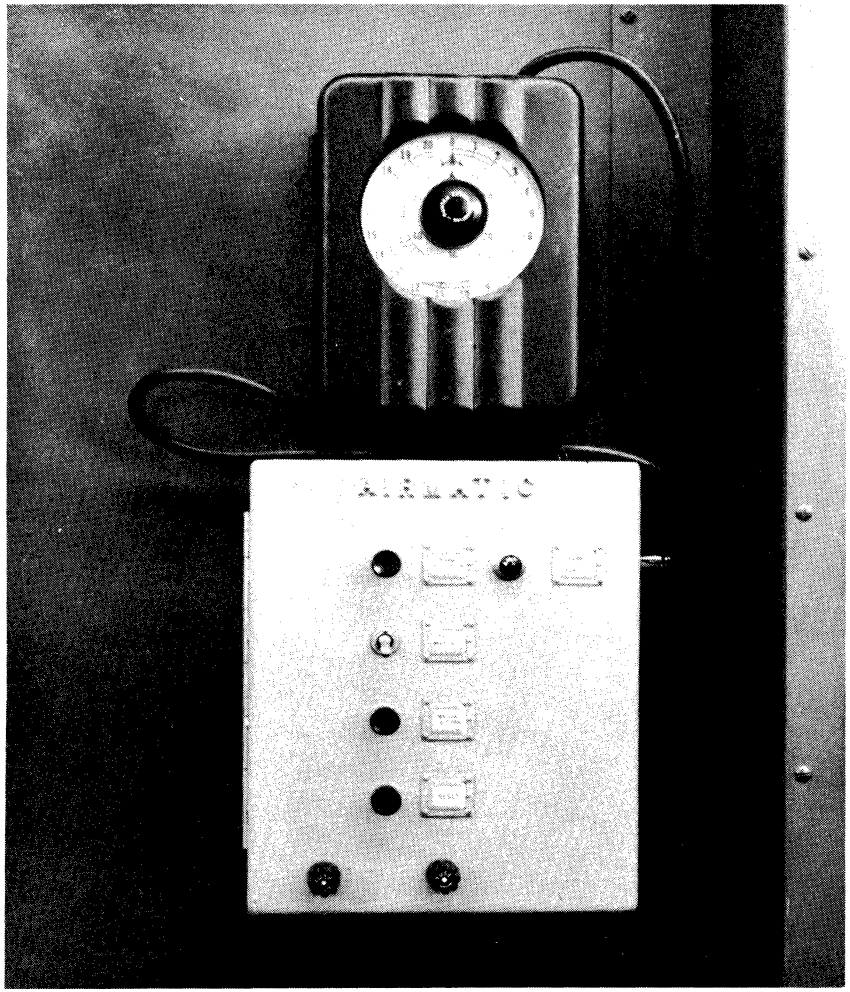


Figure 4. Rabbit Timer.

Figure 5. Interchange Box.

The remodeling should be completed by the middle of November when the pulse height analyzer is due to be delivered.

C. Chemistry Building

During the past year it finally became possible to obtain an order to air condition and control the humidity of one of the counting rooms used by this group. As a result all of the routine counting equipment is being combined with the specialized spectrometer equipment into one room (3023 Chemistry Building) which should be air conditioned by November of this year. This improvement in the counting facilities should insure reproducible counting during the humid summer months of the Michigan climate as well as during the winter.

II INSTRUMENTATION

This past year has seen little additional equipment added except for the order of the 100-channel pulse height analyzer for delivery in November. Most of the equipment described in previous reports (1-3) for work in cross section determinations and decay scheme analysis has been operating satisfactorily so that reliable experimental data could be obtained with them. The two 4π counters have been operating routinely for over a year and have been giving reproducible results.

A. 100-Channel Pulse Height Analyzer

Since the University of Michigan Reactor and the Phoenix Building present an excellent integrated facility for work with short half-lived radioisotopes some means of rapid measurement of spectra was sought. Scintillation and proportional counter detectors are already in operation in the group but they are operated through single-channel, automatic-sweep equipment which takes considerable time to sweep a spectrum. This equipment is entirely satisfactory with radioisotopes whose half-lives are long compared to the 10, 40 or 100 minute sweep times of the analyzer. It, however, limits decay scheme studies to activities with half-lives of from 10-20 minutes.

Therefore a multi-channel analyzer was sought to permit work with radioisotopes with half-lives of the order of a few minutes or possibly even a few seconds. Several different

types of analyzers were considered including 100-channel and 256-channel analyzers with computer-type memories as well as 50 or 100-channel analyzers with visual register read-out.

The latter type of analyzers were eliminated since they provided no easy way to record consecutive spectra at short intervals. Admittedly the registers could be photographed at repeated intervals during the decay of a short-lived activity, the processed film read in a microfilm reader and the decays plotted. The author has had experience with this type of operation in 1950 at Berkeley (4) and found that it left much to be desired in terms of immediate availability of the data and ease of operation.

This left the computer-type of pulse analyzers which also had some disadvantage in terms of fast read-out. The limiting factor in all presently available models proved to be the print-out time for the information stored in the magnetic core memory. Thus the tape print-out or the automatic plot print-out took the order of one second per channel to print out the data although it would be possible to dump the data from the memory in a matter of a few milliseconds or less.

Thus it would be necessary to modify one of these pulse analyzers to obtain consecutive measurements on the spectrum of an isotope with a half-life of a few minutes. Some manufacturers suggest that the memory can be broken into parts such as two 50-channel segments in a 100-channel analyzer or some even went so far as to suggest eight 32-channel segments

in a 256-channel analyzer. The complicated switching arrangements required to effect this splitting of the memory superimposed upon the already complex electronic circuitry of the analyzer seemed to warn against the use of this type of arrangement by a nuclear chemistry group.

Therefore the suggestion by the Radiation Instrument Development Laboratories of Chicago that they furnish a pulse height analyzer with duplicate 100-channel memories appeared interesting. In this instrument one memory would be storing counts coming from the sample while the other memory was plotting out the spectrum at about one point a second. At a predetermined time the memories would be switched and the second memory would start plotting out its data. In April, 1957 when this order was placed the limiting factor on the rapidity with which counts could be taken was the print-out equipment restricting the time between consecutive counts to two minutes. Since then the manufacturers have improved their print-out equipment so that the minimum interval has been reduced to about one minute.

This equipment is due to be delivered in November and should prove very helpful in measurement of short half-lived materials. (W. W. Meinke)

B. Three-inch Scintillation Crystal and Mounting

A three-inch sodium iodide(Tl) scintillation crystal has been obtained (already packaged) from the Harshaw Chemical Company for use with the 100-channel analyzer. This crystal

has been mounted with a 3-inch photomultiplier tube and will be used inside a two-inch lead shield in the specialized counting room in the Phoenix Building. It will be used alternately with a 1-1/2" standard crystal to measure spectra of different isotopes. (W. W. Meinke, R. Shideler)

III EXPERIMENTAL

The past year has seen the completion of two doctoral theses on the nuclear chemistry phase of this program, one in absolute cross section determinations and one in decay scheme analysis. Ten cyclotron bombardments were obtained between November 1, 1956 and December 7, 1956. Since that time however, work in this area has included only continued measurement of long-lived radioisotopes, and calculations and interpretations of data obtained in previous bombardments.

The program in absolute cross-sections and in decay scheme characterizations has temporarily come to a halt with the completion by Mr. Gardner and Mr. Anders of their doctoral work. It is not known at the present time whether this phase of our overall program will be continued in much detail. Few isotopes which can be obtained with the 7.8-Mev deuterons of the University of Michigan cyclotron remain to be characterized at least with the equipment available in these laboratories. Similiarly the cross-section work has proven to be very tedious and time consuming for the information obtained.

In the future much of the equipment previously used for decay scheme determinations will probably be utilized in the activation analysis and radiochemical separation program to help to discriminate the radiations of the one desired isotope from those of other possibly contaminating isotopes. Similiarly the absolute cross-section measuring equipment will probably be used in a cooperative-type program in which

charged particle cross-sections of radioactive isotopes obtained in the nuclear reactor will be determined or, vice versa, in which neutron cross-sections of radioactive isotopes obtained in the cyclotron are determined.

The program for developing optimum radiochemical separations has continued during the past year with the work on cadmium by Mr. DeVoe.

By far the most emphasis at present is being concentrated on the reactor and the activation analysis program. Several post-doctoral students are working full time on this program and other students will be working on it during the next year. Thus when the reactor finally gets into routine operation we should be able to obtain a sizeable amount of experimental information.

A. Nuclear Chemistry

1. Absolute (d,alpha) Reaction Cross-Sections and Excitation Functions

The purpose of this research was the determination of the cross sections of various (d, α) reactions induced by the 7.78 ± 0.05 Mev deuterons from the University of Michigan cyclotron. Special emphasis was given to reactions involving closed shell nuclei as target isotopes. Experimental techniques were refined to permit precision measurements of the cross section values.

The investigation involved the bombardment of thin targets of metallic zirconium, molybdenum and

titanium as well as of zinc sulfide, subsequent chemical separation of the product nuclei, identification of the products by x-ray and γ-ray spectroscopy and determination of the absolute disintegration rates of β- and x-ray emitting isotopes.

The instruments constructed during this research included: a current integrator permitting measurement of the intensity of the cyclotron beam with 1% error; two 4π β-ray proportional counters, a four-inch diameter x-ray proportional counter filled with krypton at two atmospheres pressure and a beta thickness gauge for thin films.

Procedures were developed to calibrate the current integrator, to determine, with an accuracy of 1%, the evenness of target foils by a non-destructive method, to prepare cyclotron targets of materials sublimating below 1500° C by high vacuum evaporation, to purify various reaction products by using carrier-free chemical separations with decontamination factors of 10^5 and better. Methods were devised to determine the yield of the chemical separations with radioactive tracers, to identify various isotopes by their γ- and x-ray spectra, to count absolutely x-rays and β-rays with proportional counters as well as with a coincidence set-up, and to prepare thin films used for mounting the 4π counter samples.

The absolute cross sections, σ, were measured for the following reactions:

$Zr^{90}(d,\alpha)Y^{88}$ * σ= 2.34 ± 0.28 millibarns at 7.56 ± 0.1 Mev

$Zr^{92}(d,\alpha)Y^{90}$ * σ= 3.79 ± 0.26 millibarns at 7.56 ± 0.05 Mev

$Zr^{94}(d,\alpha)Y^{92}$ * σ= 4.01 ± 0.28 millibarns at 7.56 ± 0.05 Mev

$Mo^{92}(d,\alpha)Nb^{90}$ σ= 2.95 ± 0.14 millibarns at 7.71 ± 0.05 Mev

$Mo^{97}(d,\alpha)Nb^{95}$* σ= 2.35 ± 0.14 millibarns at 7.71 ± 0.05 Mev

$Mo^{97}(d,\alpha)Nb^{95m}$ σ= 0.98 ± 0.09 millibarns at 7.71 ± 0.05 Mev

$Mo^{98}(d,\alpha)Nb^{96}$ σ= 2.53 ± 0.12 millibarns at 7.71 ± 0.05 Mev

$Ti^{46}(d,\alpha)Sc^{44}$ σ= 52.4 ± 2.6 millibarns at 7.71 ± 0.05 Mev

$Ti^{48}(d,\alpha)Sc^{46}$ σ= 28.5 ± 1.7 millibarns at 7.71 ± 0.05 Mev

$S^{34}(d,\alpha)P^{32}$ * σ= 330.3± 23 millibarns at 7.73 ± 0.05 Mev

Excitation functions were determined for the reactions with asterisks. The decay scheme of Y^{88} was investigated and a metastable intermediary state discovered. Similar metastable states were found for the decay of Mn^{54} and Sn^{113}.

Absorption curves in beryllium were experimentally determined for x-rays emitted by Y^{88} and Mn^{54}.

This work is described in detail in the project report by O. U. Anders (5). Several sections of this report are being revised for submission to THE PHYSICAL REVIEW. (O. U. Anders).

2. Systematics of (d,alpha) Reaction Yields and Possible Closed Shell Effects

The work on the absolute (d,alpha) reaction cross sections during the past several years has been aimed

at surveying these reaction yields as a function of atomic number with the hope of establishing the presence or absence of closed shell effects in these values.

One of the postulates of the Nuclear Shell Model is the relatively great stability of nuclei containing "magic" numbers of either neutrons or protons. The nuclear reaction cross section is a quantity related to the stability of the nucleus. Hughes (6) was thus able to prove the greater stability of closed shell nuclei with a plot of the capture cross sections for neutrons versus the number of neutrons as given in Fig. 6. In this plot certain isotopes such as Kr^{86}, Rb^{87}, Sr^{88}, Xe^{36}, Pb^{208}, Bi^{209}, etc., containing a magic number of neutrons, reveal significantly lower cross section values. This shows a certain inertness toward nuclear reactions for these nuclei and is an indication of their relatively greater stability.

To illustrate the same principle by means of the (p, n) reaction Blaser, Boehm, Marmier and Scherrer (7) investigated the cross sections and excitation functions for the (p, n) reactions of many target isotopes. A plot of the (p, n) cross sections which they determined for 6.7-Mev protons vs. the neutron content of the target nuclei showed the values for magic-number nuclei lying below the general trend. The values for 6.7-Mev protons, however, did not show significant deviation for all magic nuclei. Those deviating most significantly

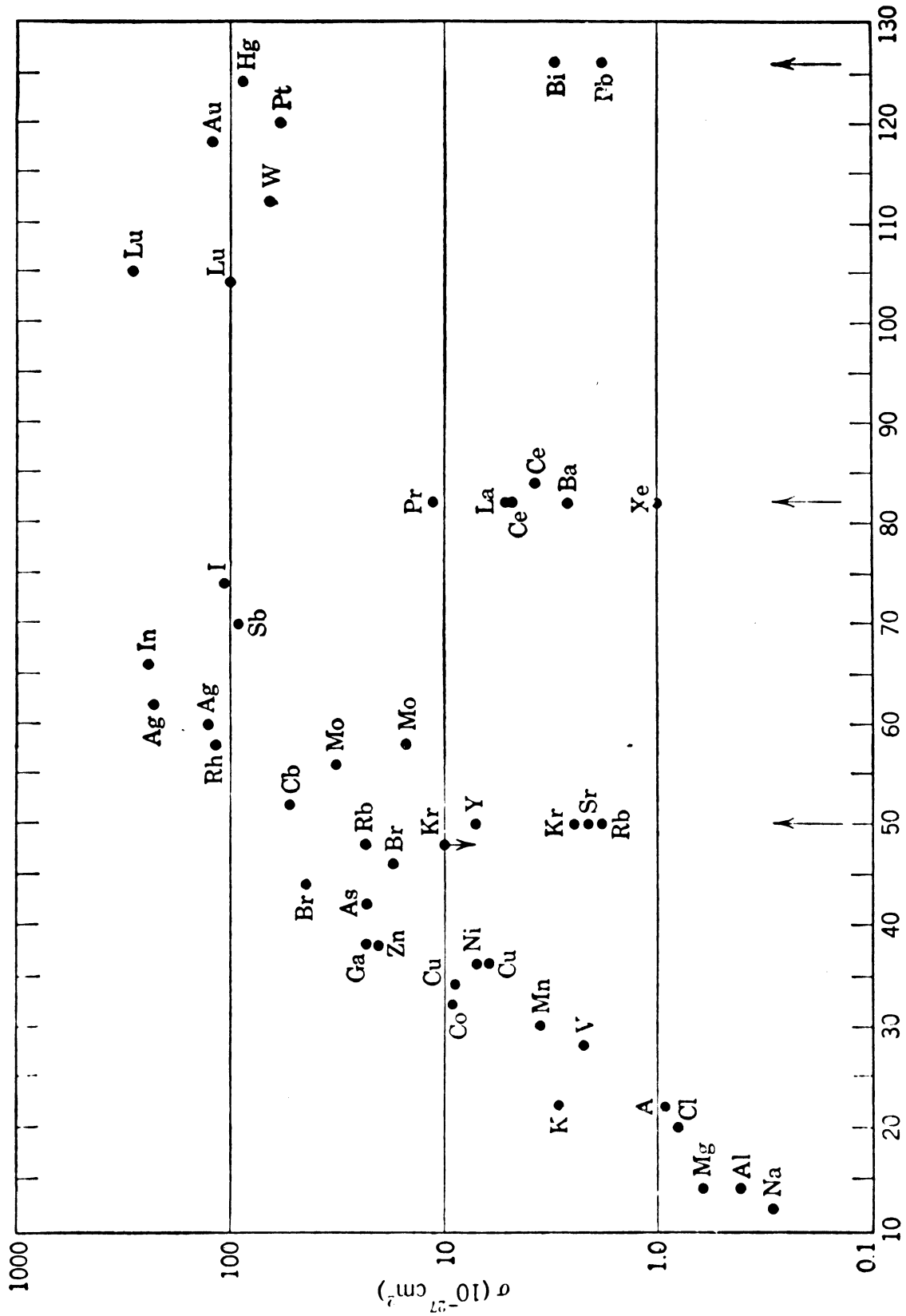


Figure 6. Capture Cross Section for 1-Mev Neutrons. (Reference 6)

in their (p, n) cross section values belonged to so called "double magic" nuclei, featuring a magic number content of both neutrons and protons.

Essentially the same type of investigation as mentioned above for the (p, n) reaction has been attempted by our group for the (d, α) reaction. It was expected that the deviation of the cross sections for magic-number nuclei would be much less significant than for the two reactions mentioned above and, that measurements giving values with an accuracy better than 10% would be required, to prove any significant difference in the cross-section values for closed-shell nuclei as compared to their non-magic neighbors. This expectation was based on the fact that approximately 2.5 times as much energy is released when the 7.8-Mev deuteron interacts with the target nucleus than in the capture of a thermal neutron. (The relatively high-energy deuterons had to be used to give sufficient yields for the nuclear reactions). This energy effect is likely to overshadow any small rest energy differences in the magic-number target nucleus which are relatively important for the (n, γ) case (8, 9).

The most significant results were expected from the comparison of the cross-section values of a nucleus having closed shells for both protons and neutrons, Zr^{90}, with those of its non-magic isotopes.

The results of this research appear to verify

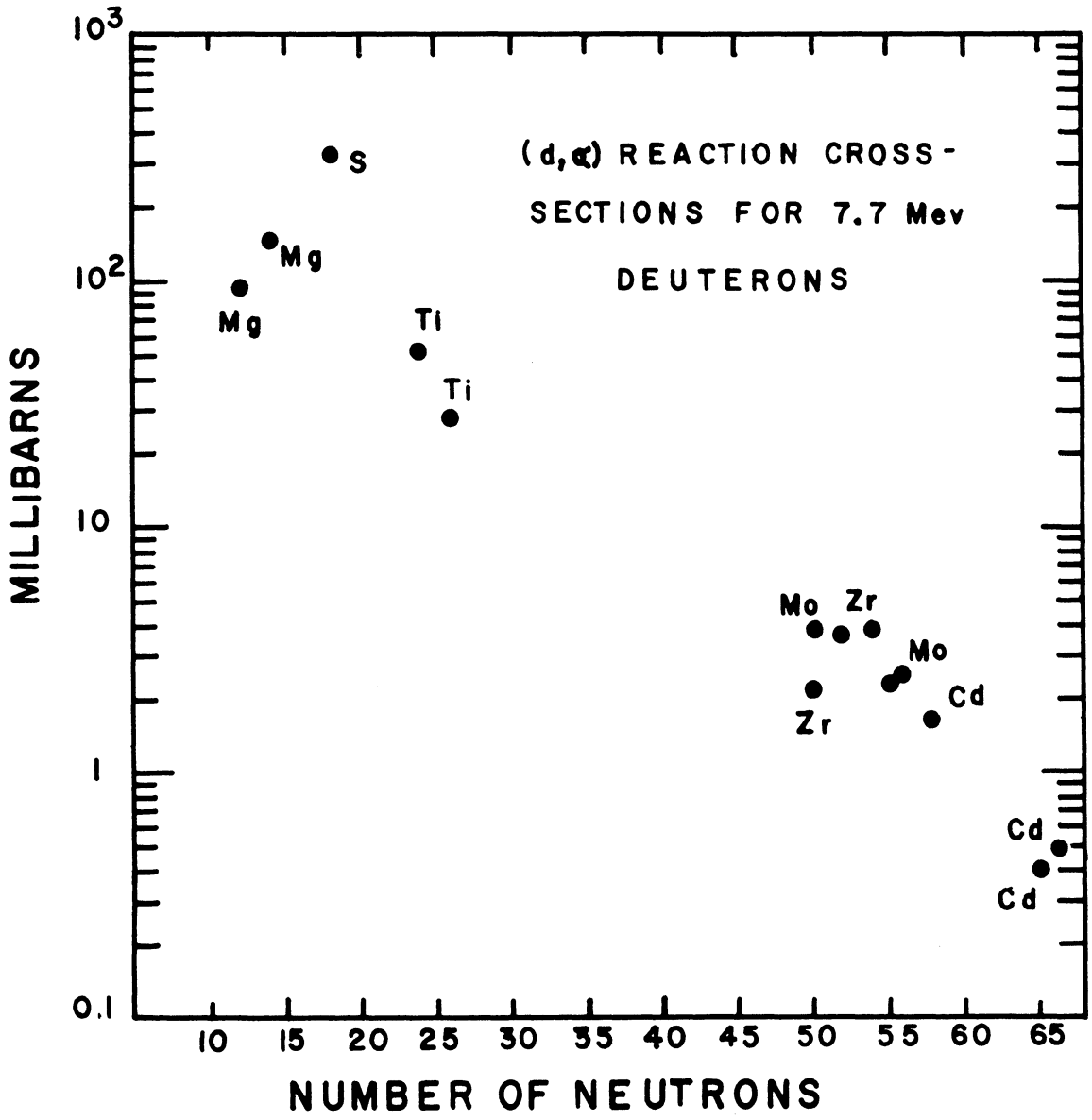


Figure 7. (d, α) Reaction Cross Sections for 7.7-Mev Deuterons.

the expectations. Figure 7 is a plot of the available (d, α) reaction cross-sections for 7.7-Mev deuterons. The values for cadmium and magnesium were determined in this laboratory by Hall (10) while the other values have been determined more recently by the author. A discussion of the errors for the points in Fig. 7 can be found in previous publications (5, 10).

Two closed-shell nuclides with a content of 50 neutrons have been investigated. While the value for Mo^{92} does not seem to deviate from the trend established by the cadmium, molybdenum and zirconium isotopes, the value for the double-closed-shell Zr^{90} is seen to lie significantly below it.

The relatively high cross-section value for sulfur is not easily explained. Although the decay curve of the phosphorus reaction product seemed to exclude any admixture of P^{33} stemming from the S^{36} $(d,\alpha n)P^{33}$ reaction, some of the P^{32} reaction product may still originate by the neutron-induced reaction $S^{32}(n, p)P^{32}$. The latter is also suspected from the curvature of the low-energy excitation function of the $S^{34}(d, \alpha)P^{32}$ reaction (5).

The trend of the (d, α) reaction cross-section values in Fig. 7 is distinctly different from that for the (n, γ) reaction found in Fig. 6. While the non-magic-number cross sections tend to greater values for increasing neutron content of the target nuclei, the

(d, α) cross sections decrease with increasing atomic weight.

Thermal neutrons do not have to overcome a potential barrier to strike the nucleus. The probability of interaction, i.e., the capture cross section, thus increases with the size of the target nucleus. In the case of deuterons as bombarding particles a certain minimum energy is required before the projectile can penetrate to the nucleus of the target atom. This threshold energy is the greater, the greater the positive charge of the target nucleus. For a given laboratory energy of the deuterons the threshold energy is exceeded more for low-atomic number nuclei than for those higher in the periodic table. The cross section, however, is energy dependent, and a steep rise of the excitation function is experienced in the vicinity of the threshold. This makes a slight excess of deuteron energy above the threshold account for a large increase of the cross sections.

To obtain a plot more closely analogous to Fig. 6, the (d, α) reaction cross sections should be plotted for those laboratory energies for which the deuterons have the same kinetic energy when reaching the nuclear surface. Such a plot would partially compensate for the effect of the Coulomb barrier, which is not encountered in the case of neutron capture.

To establish such a plot, very exact values for the cross sections must be known for the respective

ranges of the excitation functions. The energy range that could be covered by the means available for this research, however, did not suffice for such a plot. It was thus only attempted to determine the absolute cross sections for one energy, and to find some relative values of the excitation functions to determine the character of these functions.

A rather complete search for charged-particle-reaction excitation functions reported in the literature was undertaken during the initial stage of this research (11). Very few excitation functions for (d, α) reactions were found in this search, thus pointing out the need for additional data of this kind.

a) Considerations for Future Experiments

Further experiments on the (d, α) reaction cross sections may be planned as an extension of the present research. The considerations entering into the choice of the reactions should be essentially the same as the ones governing the choices for the investigation completed. (5, 10).

The energy of the available deuterons may exclude as target materials all elements for which the threshold of the (d, α) reaction is not exceeded. For the 7.8-Mev deuterons of the cyclotron of the University of Michigan this would exclude all elements with atomic number greater than about 66 (dysprosium) as was found experimentally.

The following elements had to be excluded, since the (d,α) reaction products form stable nuclei: Be, C and the odd atomic-number elements: N, F, Na, Al, P, Sc, V, Mn, Co, As, Y, Nb, Tc, Rh, In, I, Cs, La, Pm, Tb. Investigation of the (d, α) reactions of these elements would have to follow a completely different approach. This would probably include either detection of the primary α particles emitted during the reaction, or the use of a mass-spectrometer.

Of the remaining 44 elements some were excluded because the counting rates of the (d, α) reaction products would be too low to measure due to the low relative abundance of the parent isotopes and too long or too short half-lives of the products. These elements are shown in Table I.

Inability to bombard gaseous materials and count gaseous products with the equipment on hand excluded the following elements from consideration: He, Li, Ne, A, K, Kr, Rb, and Xe.

Difficulties in analyzing decay curves of the (d, α) reaction products were anticipated for a number of other elements. The critical isotopes of these elements are shown in Table II.

Several elements posed difficulties in tracing their (d, α) reaction products through the chemical separations:

No suitable tracer could be found for the

Table I. Elements Excluded from Investigation because of Half-Lives of (d, α) Reaction Products or Low Abundance of Parent Isotopes

Element	Parent Isotope	Abundance	Half-Life of (d, α) Reaction Product
B	B^{10}	18.8	4×10^{-15} sec
O	O^{18}	0.204	7.4 sec
Si	Si^{28}	92.17	10^6 years
	Si^{30}	3.12	2.3 min
Ca	Ca^{40}	96.9	7.7 min
	Ca^{42}	0.64	3.9×10^9 years
	Ca44	2.1	12.5 hours
	Ca^{46}	0.0032	22 min
	Ca^{48}	0.18	unknown
Br	Br^{81}	49.4	7×10^4 years
Pd	Pd^{106}	27.3	4.4 min, 42 sec
Ru	Ru^{99}	12.7	10^5 years
	Ru^{100}	12.7	10^4 years
	Ru^{101}	17.0	2×10^5 years
Ag	Ag^{109}	48.6	7×10^6 years
Eu	Eu^{153}	52.2	80 years

Table II. Elements Excluded from Investigation because of Decay Curve Analysis Problems

Element	Parent Isotope	Half-Life of (d, α) Reaction Product
Sn	Sn^{112}	5 hours, 66 min
	Sn^{114}	21 min, 2.5 sec, 14 min
	Sn^{115}	1.73 hours
	Sn^{117}	4.5 hours
	Sn^{118}	54 min, 13 sec
	Sn^{119}	1.9 hours, 1.1 hours
Te	Te^{120}	3.5 min, 5.1 hours
	Te^{122}	5.8 days, 17 min
	Te^{124}	3.5 min, 2.8 days
	Te^{126}	21 min, 1.3 min, 60 days
	Te^{128}	9 hours, 28 days
	Te^{130}	1 hour
Nd	Nd^{146}	17 min
	Nd^{148}	24 min
Sm	Sm^{147}	25 years
	Sm^{148}	2 years
	Sm^{149}	2.6 years
Gd	Gd^{152}	14 hours
	Gd^{154}	9 hours, 13 years
	Gd^{156}	16 years
	Gd^{157}	1.7 years
Ba	Ba^{134}	6.2 days
	Ba^{138}	13 days

Cl^{37}(d, α)S^{35} reaction since all radioactive sulfur isotopes, but S^{35}, have half-lives below 10 minutes.

Difficulties were anticipated in procuring tracers for the (d,α) reactions on Zn, Ge, Ce, and Pr. As tracer for the Zn(d, α)Cu reactions the 61-hour Cu^{67} should be used. This isotope can be obtained either by the Zn^{67}(n, p)Cu^{67} reaction or the Zn^{70}(d, αn)Cu^{67} reaction, but both would necessitate the use of isotopically enriched targets. The Ge(d, α)Ga reactions may be traced by the 14.1-hour Ga^{72} which would have to be produced by a preliminary bombardment of germanium. For the Ce(d, α)La reaction no good tracer is available. La^{141} with a 3.8-hour half-life may serve to trace the Ce^{142}(d, α)La^{140} reaction but its procurement by the low-yield Ba^{138}(α, p)La^{141} reaction is difficult and close timing of the bombardments has to be observed to make use of it.

Without use of enriched Ce^{140} no pure tracer Ce^{141} can be obtained which would be necessary to trace the Pr^{141}(d, α)Ce^{139} reaction. The use of separated isotopes was, however, avoided in the present research due to their high cost and lack of availability.

(d, α) reactions involving Mg, Cd (10) and Cr (12) had been investigated by previous workers, so that the final choice had to be made from the ten remaining elements.

The cross sections of the (d, α) reactions on iron

were not measured since the thin, high-purity iron foil necessary could not be obtained during the course of the investigation. Nickel bombardments producing 77-day Co^{56}, 71-day Co^{58} and 5.2-years Co^{60} were not performed, although thin targets could be produced by electro-deposition. The bombardment times necessary for sufficient amounts of reaction products would have been relatively long and the resolution of the decay curves of the products difficult.

Gallium and selenium containing no magic-number isotopes were of relatively small interest for an investigation of the effects of nuclear shell structure on the (d, α) reactions.

This left only S, Ti, Zr, Mo, Sr, and Sb. The (d, α) reaction cross sections of the first four were investigated in the present research.

Improvements of the data obtained in this research could be made by employing the various techniques developed for tracing the reaction products more methodically. In this connection it should be kept in mind that the method using a reference sample is independent of the knowledge of the decay constants of the tracers as well as of the reproducibility of a given counter over a long period of time.

The determination of the self-absorption of β rays in the 4π sources may be studied in order to improve some of the errors quoted. Finally, the analysis of

the decay curves may be facilitated by the use of a computer, making the resolution of many-component decay curves quicker and less laborious. This would also permit the resolution of decay curves involving isotopes whose half-lives differ by less than 50 percent.

Nevertheless, it is believed that the method of determining the (d, α) reaction cross sections has by now been sufficiently developed to obtain data precise enough for theoretical treatment. The work should now be expanded to include various other nuclides to permit a good quantitative correlation of the trends in the (d, α) reactions. More excitation functions should be measured which could be normalized to the absolute points.

The techniques developed in the present research are applicable without alteration to the study of the (d, n) reaction and, with little improvement on the chemical separations, for all reactions for which the product is different than the target element. For such reactions more attention has to be given, however, to effects of side-reactions having the same product. (O. U. Anders).

3. Nuclear Decay Scheme and Characterization Studies of (d, alpha) Reaction Products

The objectives of this work were to develop the necessary instrumentation, chemical procedures and aids in data analysis so that nuclear characterization studies

of certain (d, α) reaction products could be made.

A study was made to assess the value of a fluorescent plastic detector for use in β-ray spectroscopy. This led to the design, construction and evaluation of a hollow-type scintillation β-ray spectrometer. The performance of the device was evaluated with respect to such aspects as back-scattering from sample backing and sample holder, thick samples, geometry of detector, complex and forbidden spectra, and the useful energy range. A comparison was made with the conventional flat-type scintillator. When corrected for instrument resolution, the hollow-type spectrometer enables β-spectral distributions to be accurately studied down to energies of less than 0.1 Mev. Other instrumentation improvements include a photographic method of obtaining coincidence information.

Existing methods for the analysis of complex radioactive decay curves were studied, and a resolution program was written for the IBM 650 digital computer using the method of weighted least squares. A new method of solution, based on a Fourier analysis, is proposed which seems to have advantages over all previous approaches. Here prior knowledge of the number of components in the decay curve is not needed since it automatically appears in the solution, and the calculation is not endangered by the presence of half

lives of similar value. Regarding β-spectral analysis, one computer program was written to provide a Kurie analysis, and a second for general linear curve fitting using the method of unweighted least squares.

Characterization studies were made of the isotopes Co^{62}, Y^{92}, Ir^{196}, and Sc^{47} which were produced by the (d, α) reaction in cyclotron-bombarded targets. Isotopically enriched samples were used with the Co^{62} and Ir^{196} studies. Chemical separations were required in all cases, and carrier-free procedures were developed for cobalt, yttrium, and scandium. New decay schemes are proposed for Co^{62} and Y^{92}, and are substantiated by arguments based on nuclear shell structure theory. The previous assignment of an ~ 8-day activity of iridium to Ir^{196} is shown to be incorrect, the activity being due rather to Ir^{189} and Ir^{190}. New γ-ray information is given for Ir^{189} and Ir^{190}, together with excitation functions for the (d, α) reaction on Pt^{194} and Pt^{196} for deuteron energies in the range of 9.6 to 20.4 Mev. The γ-ray information obtained from the scandium work verifies the data reported recently by other workers.

This work is described in detail in the project report by D G. Gardner (13). A paper on the decay scheme of Co^{62} will be published in the September 15, 1957 issue of THE PHYSICAL REVIEW. Papers on Y^{92} and Ir^{196} decay schemes and on the Hollow Scintillator Spectrometer have been submitted for publication.
(D. G. Gardner)

4. Nuclear Data Analysis: Application of Digital Computers

This subject is covered in detail in the report by D. G. Gardner (13). The use of digital computers in Kurie analysis of beta spectrum has been reported in the previous progress report (1).

Considerable additional work has gone into an exploration of a possible system for obtaining half-life and intercept values with appropriate statistical errors from multi-component decay curves. A number of methods reported in the literature for doing this have been reviewed (13) and their limitations discussed.

A method for the analysis of decay curve type problems, based on the Fourier transform has been proposed. The results appear as a frequency spectrum of $g(\lambda)$ vs. λ . Fundamental to the entire analysis is the assumption that, while in theory $g(\lambda)$ could be represented by a discontinuous set of Δ functions, it is impossible to measure $g(\lambda)$ exactly by an experimental means and so, due to the inherent error in the data, $g(\lambda)$ lapses into a continuous function. However, it is also true that the resolution of the peaks in $g(\lambda)$ may be made as fine as desired merely by extracting an increasing number of values of $f(x)$ from the initial data, and refining the integration scheme accordingly.

An error analysis is available for the integration scheme and also for the cut-off error. The error in the original data plus the error in the numerical

calculations due to rounding off numbers, etc., will produce peak profiles showing a normal distribution as long as the cut-off error is very small and the integration scheme employs small enough intervals. Therefore, a standard deviation may be obtained from the peak itself. Under favorable conditions, the standard deviation should not change greatly from peak to peak in a multicomponent system, and hence may be used as a test to see if a peak might contain more than one component for λ's extremely close together. That the number of components present may be determined by counting the number of peaks in the results is one of the best features of this method.

A fairly large computer will be required to handle the problem if the maximum resolution inherent in the method is to be approached. However, the computer program itself should not be logically complex. Most of the subroutines needed, for example the determination of sine, cosine, exponential, logarithm, and routines for simple integration and interpolation, are available at most computer installations. Only the subroutines for the determination of the complex function, and Filon's integration scheme would then be needed.

Besides the fact that the number of components falls out of the analysis, it is felt that this method has certain other advantages over previous methods. For example, the restriction to equal intervals is merely

a requirement of the particular integration scheme used, and is not basic to the procedure as in Cornell's, Householder's, and Prony's methods. Human judgment in the resolution process has been reduced to roughly estimating the spread in half-lives so that a minimum of computational time may be used. This is an advantage over all previous methods. The fact that errors entering the program at different places may be estimated individually may prove advantageous. Finally, the numerical computation itself is not as complicated as in Prony's or Householder's method, and the occurence of two half-lives very close together does not endanger the entire calculation as it does in the former methods. Further elaborations on this method are available (13). (D. G. Gardner)

5. Chemical Procedures

In this report we continue the practice observed previously of presenting specific radiochemical separation procedures developed in these laboratories. Some of these differ only slightly from procedures reported elsewhere but they are included because they have been designed for our specific requirement and may be of some use to others in designing an experiment. They are written in the form used by Meinke in his compilation of procedures (14).

CHEMICAL SEPARATIONS

Element separated: P (carrier-free) Procedure by: Anders

Target Material: ZnS Time for sep'n: 6 hrs.

Type of bbdt: ~7.8 Mev deuterons Equipment required: Pyrex beakers (800 ml); 1 Phillips beaker (250 ml); centrifuge; pH meter; 2 centrifuge tubes, 50 ml; Erlenmeyer flask, 1000 ml; 3 ion exchange columns, 25 cm long, 1 cm diameter.
Dowex 50 resin (100-200 mesh); $FeCl_3$ solution (1/2 saturated); bromine; carbon tetrachloride; conc. HNO_3, H_2SO_4; NaOH(pellets); Na_2CO_3 powder; conductivity water; 30% H_2O_2.

Yield: ~70%

Degree of purification: 10^4

Procedure:

(1) Place target into 250 ml. Phillips beaker containing the P^{33} tracer plated on Zapon film. Add a mixture of 20 ml. CCl_4 and 5 ml. Br_2 and 5 ml. conc. HNO_3. Permit to react for 1/2 hour, shake at intervals. (Note 1)

(2) Heat gently and evaporate to near dryness, shake occasionally.

(3) Add 3 - 4 ml. conc. H_2SO_4 to dissolve the Mylar (Polyester film) and the Zapon film of the tracer sample. Don't heat. (Note 2)

(4) After clear solution is obtained, add water to hydrolize Mylar. Centrifuge off the precipitate. Decant and save supernate.

(5) Add a NaOH pellet with a drop of water. The precipitate dissolves with little residue. Dilute, centrifuge and add supernate to that of step 4. (A milky solution results.) Treat residue with a few drops H_2SO_4 and add it to supernates. (Note 3)

(6) Centrifuge for 4 minutes to get rid of the colloidal terephthalic acid. Add supernate into a 1 liter Erlenmeyer flask and dilute to approximately 500 - 600 ml. (depending on how much H_2SO_4 had been used).

(7) Pass the solution through a cation exchange column charged with 10 ml. 100 - 200 mesh Dowex-50 in hydrogen form. The metallic ions are retained by the resin.

(8) Evaporate eluate to less than 500 ml. Neutralize to pH 6.5 with less than 4 gr. solid Na_2CO_3 anhydr. Heat while adjusting pH.

(9) Pass solution slowly through a Dowex-50 $Fe(OH)_3$ column that has been prepared as described below. Wash with 50 ml. H_2O. Phosphorus is retained by the column. (Note 4)

(10) Elute phosphorus activity from column with 100 ml. of 0.125 N NaOH and pass it directly through a Dowex-50 column in its hydrogen state. Wash columns with 20 ml. H_2O.

(11) Evaporate eluate to near dryness. Destroy dissolved column resin (~1 mg) with a few drops 30% H_2O_2 and plate for counting after H_2O_2 has been destroyed by gentle heating.

Notes:

(1) The solution of Br_2 in CCl_4 oxidizes the sulfide to sulfate and avoids formation of colloidal sulfur which would interfere with attaining an initial homogeneous solution. Escape of phosphine is also avoided in the oxidizing medium.

(2) Phosphoric acid is volatile when heated to the fuming temperature of sulfuric acid.

(3) A homogeneous solution of the terephthalic acid monomers results in the alkaline solution. It is expected that only a negligible amount of P^{32} is carried by the reprecipitating acid before isotopic exchange with the P^{33} tracer has occurred. Steps 8, 9, 10 form a unique method of separating phosphorus activity from sulfur.

(4) The Dowex-50 $Fe(OH)_3$ column was prepared by the method given by McIsaac and Voigt. Dowex-50 (100 - 200 mesh) obtained from Bio . Rad Company, Berkeley, California, was filled into the column and washed with distilled water. Conc. HCl and again distilled water was passed through the column until eluate did not change red litmus. A solution 1 N in $FeCl_3$ and 0.1 N in HCl was then passed through the column until eluate turned yellow. It was then rinsed with water to free it from excess iron. 50 ml. of 2 N NH_4OH was passed through next, turning the resin purple in color. The column was finally rinsed with distilled water to remove excess ammonia.

CHEMICAL SEPARATIONS

Element Separated: Sc (carrier-free) Procedure by: Anders

Target Material: Ti Time for sep'n: ~4 hrs.

Type of bbdt: ~7.8 Mev deuterons

Yield: ~50%

Degree of purification: $\sim 10^5$

Equipment required: 4 Pyrex beakers (50 ml); centrifuge; centrifuge tubes 40 ml., 15 ml.; platinum wire 6 inch; Erlenmeyer flask 250 ml.; medicine droppers; glass frit filter funnel, coarse; Whatman 42 filter paper; micropipet 50λ; small ion-exchange column.
Dowex-2 resin (200 - 400 mesh); conductivity water; conc. H_2SO_4, HNO_3, HCl, NH_4OH; 30% H_2O_2; NaOH (solid pellets); $NaHCO_3$ (saturated solution); $KClO_3$ (crystals); HCl-gas tank.

Procedure:

(1) Place tracer scandium in small beaker, add Mylar substrate. Add 2 ml conc H_2SO_4 plus a few drops of 30% H_2O_2. Add bombarded titanium foil, heat until dissolved. Oxidize purple solution with few drops of HNO_3 (some TiO_2 precipitates at this point, if solution is too hot, but this does not interfere). (Note 1)

(2) Transfer to 50 ml centrifuge cone and precipitate $TiO_2 \cdot aq$. with several pellets of NaOH. Wash twice.

(3) Dissolve with conc HCl. (If solution is not complete, try adding water and heat gently). Centrifuge and transfer supernate to another centrifuge cone.

(4) Add slowly a saturated solution of $NaHCO_3$ until initial precipitate still dissolves on stirring. (Note 2)

(5) Heat gently to effect homogeneous precipitation of $TiO_2 \cdot aq$. Do not permit the pH to rise above about 5.5.

(6) Repeat steps 3, 4 and 5 and combine the supernate with that of step 5.

(7) Saturate combined supernates with HCl gas (Cool!). Centrifuge off the NaCl.

(8) Evaporate to 5 ml and repeat step 7.

(9) Add a crystal of $KClO_3$, shake and transfer to a small anion exchange column charged with 1 ml. Dowex-2 resin which has been saturated with conc. HCl containing a few mg $KClO_3$ per 100 ml.

(10) Permit to absorb. When liquid level reaches the resin bed add five drops of conc. HCl with $KClO_3$ and permit again to reach the bed-level. Elute the scandium activity with approximately 12 ml conc HCl-$KClO_3$. (Check with monitor when activity is eluted).

(11) Pour the eluate rapidly into a 250 ml. Erlenmeyer flask containing 30 ml. of a solution made of 11 parts 8 N NH_4OH and 1 part 30% H_2O_2. Shake vigorously. (Note 3)

(12) After fumes subside, cool the clear solution and pass it twice through a double-layer of Whatman 42 filter paper positioned over a glass-frit funnel. (The scandium activity remains on the filter in radio-colloidal form).

(13) Wash filter paper with 10 ml. of the mixture of step 11 to which about 3 ml. conc. HCl has been added.

(14) Wash with 5 ml. alkaline distilled water followed by a wash with 5 ml. conductivity water.

(15) Elute the scandium activity from the filter paper by passing 5 ml. of 4 M HCl twice through the filter paper (some of the activity will still remain on the paper, but most of it will be eluted).

(16) Evaporate to near dryness and plate for counting.

Notes:

(1) The Mylar is dissolved first, since titanium forms a precipitate in hot conc. H_2SO_4 solution containing peroxide.

(2) $Ti(OH)_4$ is precipitated homogeneously from a solution of pH 2, while the $Sc(OH)_3$ precipitates only from a pH of 7.

(3) A sudden increase of the pH in a strongly oxidizing medium transforms any titanium present into the titanate ion whose ammonium salt is soluble, while the trace amount of scandium, being insoluble in the medium "precipitates" in the form of a radio-colloid.

CHEMICAL SEPARATIONS

Element Separated: Mn (carrier-free) Procedure by: Anders

Target Material: Fe, Cu, Ag. Time for sep'n: ~10 hrs.

Type of bbdt: ~7.8 Mev deuterons

Yield: ~50%

Degree of purification: $\sim 10^4$

Equipment required: Continuous extractor; blowtorch Pyrex beaker, 200 ml., 50 ml.; filter crucible; Erlenmeyer flask, 250 ml.; porous plate; aluminum foil; 2 centrifuge tubes, 15 ml. centrifuge; platinum wire, 6 inch; 2 small ion exchange columns; medicine droppers.
Dowex-2 resin (200 - 400 mesh); conc. HNO_3, HCl, NH_4OH; conductivity water; iso-propyl ether; HCl-gas tank; iron carrier (10 mg/ml).

Procedure:

(1) Heat target in hood and free iron foil from backing plate.

(2) Place iron foil with adhering solder in beaker and add conc. HNO_3. Warm gently and permit copper and silver to dissolve. Decant green solution and store after ebullition ceases.

(3) Dissolve remaining iron foil in dilute HCl.

(4) After the iron is dissolved filter off any AgCl present, add 5 ml. conc. HNO_3 and evaporate to dark yellow syrup of about 10 ml.

(5) Transfer to bulb of continuous extraction apparatus and add 25 ml. conc. HCl and 5 ml. H_2O to make about 9 M in HCl.

(6) Place approximately 175 ml. iso-propyl ether into 250 ml. Erlenmeyer flask; add a small piece of porous plate, and connect to extractor and place condensor on top of extractor. Wrap extractor bulb with aluminum foil and place in beaker with water. Heat the Erlenmeyer flask to boil moderately and permit to extract in dark over night.

(7) Evaporate extracted aqueous solution to near dryness, take up with few ml. conc. HNO_3. Add 3 drops Fe-carrier.

(8) Transfer to 15 ml. centrifuge cone and precipitate with NH_4OH. Wash twice and dissolve with dil. HCl.

(9) Precipitate with NH_4OH and wash twice.

(10) Dissolve precipitate with conc. HCl and saturate with HCl gas.

(11) Apply solution to an anion exchange column charged with approximately 1 ml. Dowex-2 resin saturated with conc. HCl.

(12) Permit to adsorb. Add 5 drops conc. HCl and soak in slowly.

(13) Elute Mn^{54} with 12 ml. of conc. HCl.

(14) Evaporate eluate to near dryness. Take up with 9 M HCl.

(15) Apply to second column charged with approximately 1 ml. Dowex-2 saturated with 9 M HCl.

(16) Permit to adsorb. Add 5 drops 9 M HCl and soak in slowly.

(17) Elute Mn^{54} with 12 ml. 9 M HCl.

CHEMICAL SEPARATIONS

Element separated: Cobalt

Procedure by: Gardner

Target Material: Nickel Oxide

Time for sep'n: 1/2 hr.

Type of bbdt: 7.8 Mev deuterons

Equipment required: Ion exchange column of AG 2-x8, 200-400 mesh resin obtained from Bio-Rad Labs. Resin bed was 8 mm x 140 mm.

Degree of purification: Estimated $\sim 10^5$ from Ni and Cu.

Advantages: Carrier-free separation with high decontamination.

Procedure:

(1) Dissolve NiO in 5 ml of 10 N HCl. Evaporate to small volume, place in column previously washed with conc. HCl.

(2) Remove Ni from column by elution with 15 ml of 8 N HCl at rate of 1 or 2 drops/sec. Both Co and Cu remain on column.

(3) Remove Co by elution with 6 - 8 ml. of 4 N HCl. Last ml. or so possibly contaminated with small amount of Cu.

(4) Mount for counting.

Notes:

(1) General reference: Hicks, H. G., et al., "The Qualitative Anionic Behavior of Resin, 'Dowex 2'", Livermore Research Laboratory Report, LRL-65, Dec. 1953.

CHEMICAL SEPARATIONS

Element Separated: Y (carrier-free) Procedure by: Anders

Target Material: Zr Time for sep'n: ~3 hrs.

Type of bbdt: ~7.8 Mev deuterons Equipment required: 4 Pyrex beakers (50 ml.); centrifuge; centrifuge tubes 40 ml., 15 ml., (Pyrex); Lustroid tube (10 ml.); micro pipette 50; platinum wire; small anion exchange column; medicine droppers; calcium carrier (10 mg/ml); zirconium carrier (10 mg/ml). Dowex-2 (200-400 mesh); conductivity water; conc. H_2SO_4, NH_4OH, HCl, 30% H_2O_2; HCl-gas tank.

Yield: ~85%

Degree of purification: $\sim 10^5$

Procedure:

(1) Cut the tracer yttrium, deposited on Zapon film, from aluminum sample plate and place in small beaker, add bombarded zirconium foil plus Mylar substrate. Add 1.5 ml. conc. H_2SO_4 and a few drops 30% H_2O_2. Heat to fumes until dissolved (cool and add more H_2O_2 at intervals). (Note 1)

(2) Transfer clear solution to a 40 ml. centrifuge tube, add 30 ml. H_2O and precipitate zirconium hydroxide with conc. NH_4OH. Stir with platinum wire; centrifuge and wash twice.

(3) Dissolve precipitate with a minimum amount of conc. HCl, add 3 mg calcium carrier and one drop $NbCl_5$ carrier (10 mg/cm^3). Transfer to 10 ml. Lustroid tube.

(4) Precipitate CaF_2 (and YF_3) with 3 ml. conc. HF. Centrifuge (water in centrifuge cups!) and wash twice.

(5) Transfer precipitate to a 15 ml. centrifuge cone, centrifuge, decant, add 0.5 ml. conc. H_2SO_4.

(6) Heat to fumes to drive off HF. Cool. Add 3 mg. Zr-carrier and dissolve residue in 10 ml. warm water.

(7) Precipitate zirconium hydroxide with conc. NH_4OH. Wash three times with conductivity water. (Note 2)

(8) Dissolve precipitate with a few drops conc. HCl and saturate with HCl gas.

(9) Transfer solution to a small anion exchange column charged with 1 ml. Dowex 2 resin saturated with conc. HCl.

(10) Adsorb zirconium onto the resin slowly. After liquid level reaches resin bed add 5 drops of conc. HCl and permit to soak in. (Note 3)

(11) Elute carrier-free yttrium reaction product with 5 ml. conc. HCl at a rate of 1 drop in 7 seconds. (Note 4)

(12) Collect eluate when activity starts coming through. Evaporate to near dryness and plate for counting.

Notes:

(1) A homogeneous solution is obtained under rather severe conditions. Complete isotopic interchange between the Y^{88} tracer and the reaction product is thus accomplished.

(2) This washing removes the calcium carrier and must be done thoroughly, if carrier-free yttrium is to be obtained.

(3) Try to wash the walls of the column free of activity with these 5 drops.

(4) The yttrium is not absorbed by the resin in hydrochloric acid but is easily eluted, while zirconium and niobium are strongly absorbed from a conc. HCl medium.

CHEMICAL SEPARATIONS

Element Separated: Nb(carrier-free) Procedure by: Anders

Target Material: Mo Time for sep'n: ~4 hrs.

Type of bbdt: ~7.8 Mev deuterons Equipment required: Pyrex beakers (50 ml.); centrifuge; centrifuge tubes 40 ml., 15 ml.; medicine droppers; micropipette 50λ; platinum wire 6 inch; small anion exchange column; molybdenum carrier (10 mg/ml); aluminum carrier (10 mg/ml). Dowex-2 (200-400 mesh); conductivity water; conc. H_2SO_4, NH_4OH, HCl, HNO_3; 30% H_2O_2; HCl-gas tank.

Yield: ~50%

Degree of purification: $\sim 10^5$

Procedure:

(1) Place tracer niobium in small beaker, add bombarded molybdenum foil plus Mylar substrate. Add 30 drops of conc. H_2SO_4 and a few drops H_2O_2. Heat to fumes until dissolved. (Cool and add H_2O_2 together with a few drops of conc. HNO_3 at intervals.) (Note 1)

(2) Transfer clear yellow-brown solution to 40 ml. centrifuge cone, add 30 ml. H_2O and 2 mg aluminum carrier. Stir well. Make alkaline with conc. NH_4OH. Centrifuge and wash twice. (Note 2)

(3) Dissolve precipitate with a minimum of conc. HCl, dilute and reprecipitate with conc. NH_4OH. Centrifuge and wash twice.

(4) Dissolve precipitate in conc. HCl, add few drops of molybdenum carrier, dilute and reprecipitate with conc. NH_4OH. Centrifuge and wash twice.

(5) Dissolve precipitate in minimum amount of conc. HCl and saturate with HCl gas. (Note 3)

(6) Transfer this solution to a small anion-exchange column charged with 1 ml. Dowex-2 resin saturated with conc. HCl.

(7) Permit to absorb slowly. When liquid level reaches the resin bed add five drops conc. HCl and try to wash glass walls with this. Permit to soak in and add about 1 ml. conc. HCl.

(8) Elute the aluminum plus some salt (NH_4Cl) with 12 ml. of 7 M HCl. (Note 4)

(9) Elute carrier-free niobium with 25 ml. of 4 M at a rate of 1 drop in 5 - 7 seconds.

(10) Evaporate the niobium fraction to near dryness and plate for counting.

Notes:

(1) Complete mixing of tracer and reaction product is achieved in the homogeneous solution. All elements are oxidized to their highest valence state.

(2) Ammonium molybdate and ammonium pertechnate are soluble and thus not precipitated together with the aluminum.

(3) During most bombardments the precipitation of salt, be it $AlCl_3$ or NH_4Cl, was observed when the solution was saturated with HCl. This, however, did not interfere with the ion-exchange step.

(4) The specifications given in the procedure have to be rigidly observed as they are rather critical.

CHEMICAL SEPARATIONS

Element Separated: Nb (carrier-free) Procedure by: Anders

Target Material: Zr^{95} Time for sep'n: 4 hrs.

Type of bbdt: ~7.8 Mev deuterons Equipment required: 2 Pyrex beakers (50 ml.); centrifuge; 2 centrifuge cones 15 ml.; medicine droppers; platinum wire 6 inch; small anion exchange column; zirconium carrier (10 mg/ml). Dowex 2 resin (200-400 mesh); conductivity water; conc. H_2SO_4, HCl, NH_4OH; 30% H_2O_2; HCl-gas tank.

Yield: ~50%

Degree of purification: 10^5

Procedure:

(1) Destroy oxalate complex with 1 ml. conc. H_2SO_4 plus 3 drops 30% H_2O_2.

(2) Dilute with 10 ml. H_2O and precipitate zirconium-hydroxide with conc. NH_4OH. Centrifuge, wash once. Repeat step 2 once more.

(3) Dissolve precipitate with minimum amount conc. HCl, saturate with HCl gas and apply to small anion exchange column charged with 1 ml. Dowex 2 which had been saturated with conc. HCl.

(4) Absorb slowly. When liquid level reaches the resin bed add 10 drops conc. HCl and try to wash sides of column wall. Permit to soak in.

(5) Elute elements like alkaline earths and zirconium with two 5 ml. portions of 7 M HCl.

(6) Elute Nb^{95} tracer with 7 ml. of 2 M HCl made up with conductivity water. Discard the first ml. and use rest as tracer.

B. Radiochemical Separations

1. The Development and Evaluation of Radiochemical Separation Procedures for Barium, Strontium, Calcium, Silver and Indium

The experimental work for this investigation was completed almost two years ago (1-3) but the problem of suitable publication form was not solved. Several manuscripts had been submitted to ANALYTICAL CHEMISTRY from July 1956 to December or January 1957 covering parts of this work. Discussions with the editors indicated that considerable condensation was required and that they preferred to publish all four papers as one paper. The revisions, condensations and combinations have been made and the galley proofs were checked in early October. This paper then should be published in ANALYTICAL CHEMISTRY in either the November or December 1957 issue.

Now that the format for the separation papers has been established the indium work will be written up for publication. (D. N. Sunderman, W. W. Meinke)

2. Further Indium Separations

In the previous progress report (1) it was pointed out that indium separations by H_2S precipitation of indium sulfide, by solvent extraction of the bromide with diethyl ether, and by anion exchange in hydrochloric acid solution had been evaluated. The extraction of indium into a TTA-benzene solution both with 10 milligrams

of indium carrier present and carrier-free has been tested as a further possible separation procedure. This chelating agent (TTA) has been used for separation of a number of transuranium elements and also has occassionaly been used in fission product separations.

Figure 8 summarizes the yield data obtained for indium extraction into three different concentrations of TTA and benzene. Standard buffer solutions were used to rigidly control the pH; the pH given in the graphs is the pH at the end of the extraction. It can be seen that for the 0.1 M TTA solution there is very little difference between the carrier and carrier-free separation, although with the more concentrated solution there is some difference.

Preliminary evaluation of the decontamination afforded by this method at a pH of 2.1 and a TTA concentration of 0.5 M gives the values shown in the last column of Table III. Further data for this work is being accumulated now and will be published in the near future. (I. Ackermann, W. W. Meinke)

3. Cadmium Separations

In view of the interest in the reactor industry in burn-up determinations for all types of reactors we have decided to continue the separation work with cadmium. Cadmium, like silver is in the valley of the fission product distribution but becomes important in fast

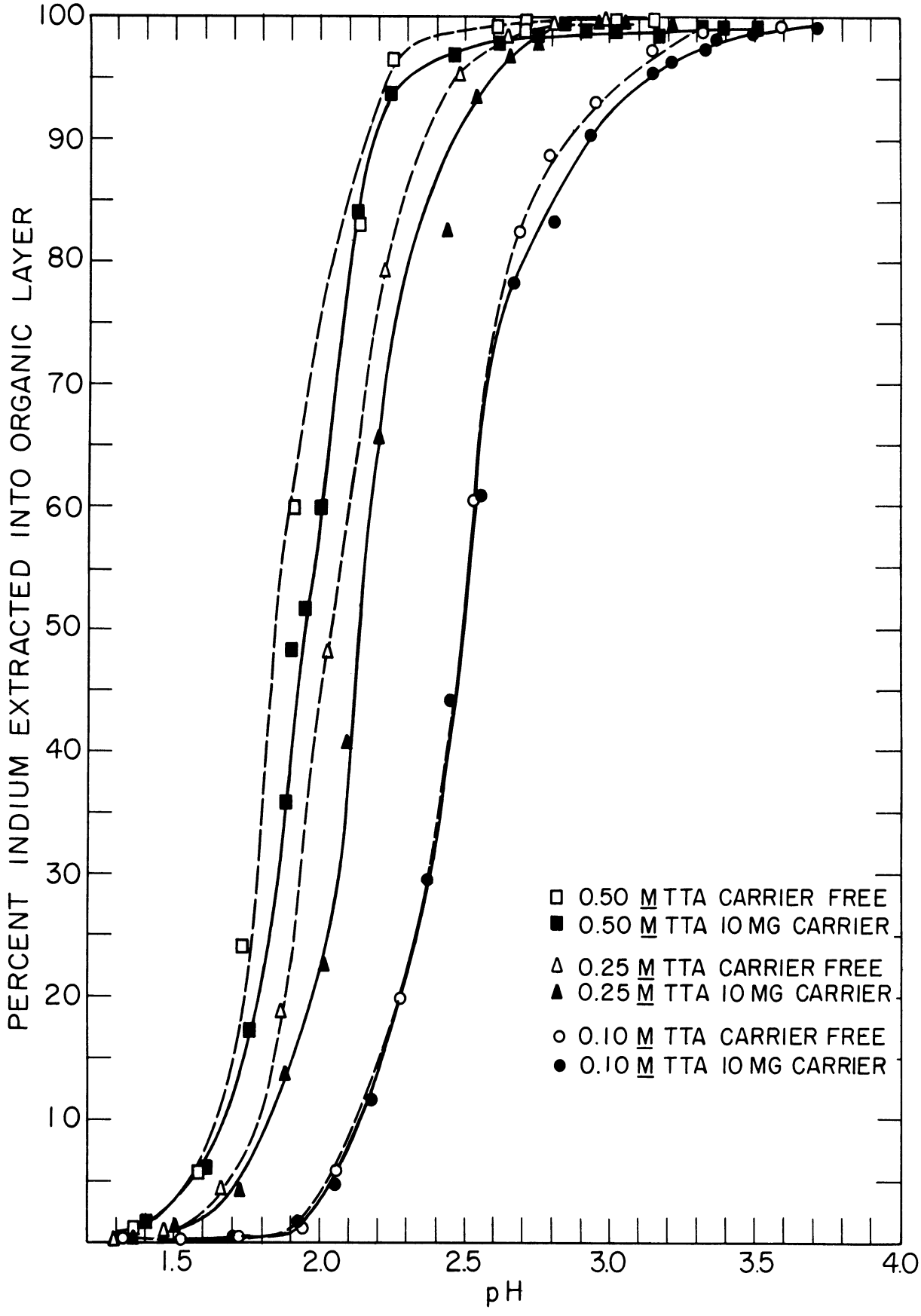


Figure 8. Yield Data For Indium Extraction Using TTA-Benzene.

Table III. Yield and Contamination Data for Indium Separation Methods

Method	H_2S Pptn.	Extraction	Ion Exchange	TTA Extraction (0.5M) pH 2.1
Element	Percentage carried with the indium			
Antimony	96.	2.2, 0.25*	2.0	0.1
Cerium	20-70 (var.)	0.01	0.13	0.3
Cesium	2.0	0.015	0.11	0.05
Chromium	1.0	0.012	0.6	0.6
Cobalt	1.7	0.009	43.	0.5
Indium	91.8±2.5	93.0±2.0	95.5±1.2	89.4
Iodine	4.3	6.7, 6.8*	1.1	-
Iridium	4.9	0.017	3.7	0.35
Ruthenium	96.	0.015	22.	0.7
Selenium	80.	23., 7.*	0.3	0.1
Silver	100.	0.055	25.	1.0
Strontium	100.	0.006	0.1	0.5
Tantalum	1.6	0.08	6.2	3.1
Tin	97.	48., 40.*	0.4	-
Zirconium-Niobium	8.7	0.018	31.	49.5

* These values were obtained with a procedure employing two 4.5 M HBr washes.

reactors in evaluating burn-up.

Therefore a critical evaluation of radiochemical separation procedures for cadmium was begun. The procedures reported in the literature were collected and subdivided into individual separation steps. Those steps which were found particularly adaptable to radiochemical separations were studied experimentally to determine optimum conditions for separation. These procedures were further evaluated using optimum conditions to determine the degree of separation from a number of representative elements.

a) Literature survey

The most widely used method of separation for cadmium reported in the literature involves the use of lanthanum hydroxide, iron hydroxide and indium hydroxide scavenges with the subsequent separation of cadmium as the sulfide. This type of method has been investigated previously in conjunction with the separation of indium (1) and hence was not explored further.

Several extraction methods have been used, e.g., the oxine in chloroform, 5% pyridine in chloroform, dithizone in carbon tetrachloride, and the complex iodide in ethyl ether. The dithizone and iodide methods will be evaluated in the future.

Inorganic precipitants utilized include $Fe(CN)_6^{-4}$ as well as various reducing agents to precipitate elemental cadmium, none of which are very selective. The

most specific of these precipitants is the Reinecke salt (15) (see below) which is evaluated here.

There is also a wide variety of organic precipitants most of which are not at all specific or selective, and therefore are of limited use for a general separation procedure. One method that uses 2-(o-hydroxyphenyl) benzoxazole (16) seemed more specific for cadmium than the others and is evaluated here.

Little radiochemical use has been made of the electrochemical separation of cadmium in a quantitative separation although much has been done on electroplating. A procedure of this type will be evaluated in the future.

b) Experimental

An evaluation of the decontamination of cadmium has been made for the Reinecke salt precipitate $\left[Cd(thiourea)_2\right]\left[Cr(CNS)_4(NH_3)_2\right]_2$ and 2-(o-hydroxyphenyl) benzoxazole. Since cadmium is very difficult to separate from zinc, the initial conditions were adjusted to obtain the optimum separation for this couple. Care was taken to assure that the yield of cadmium was greater than 50% during these experiments.

The optimum conditions for the cadmium-zinc separation included a 1% thiourea solution at a pH of 2.2, 2.5 ml of 4% ammonium reineckate monohydrate in water was used as a precipitating agent and the precipitate was washed once in 5 ml of 1% thiourea solution.

Preliminary contamination data (Table IV) for the cadmium reineckate precipitation at two acidities show that two ions which contaminated the separation at pH 2.2 did not contaminate in 1 N HCl. These ions are those which hydrolyze in an acid solution of pH 2.2.

The conditions used for the organic precipitate (HPBZ) include a pH of 13, a 15% ammonium tartrate solution, and 5.5 ml of 1% HPBZ in 95% ethyl alcohol. This precipitate is very flocculent causing occlusion of the contaminating nuclides, while the reineckate is very crystalline, and forms more slowly. This difference is readily seen in the preliminary contamination values shown in Table IV. (J. DeVoe)

4. Use of Molecular Sieves For The Decontamination of I^{131} From Air

A series of small-scale laboratory tests were conducted to determine the feasibility of the decontamination of iodine vapors from air by means of adsorption on a highly efficient solid adsorber, the so-called molecular sieve (17). A properly developed process of this type could offer considerable advantages over conventional equipment utilized for environmental protection by groups concerned with the disposal of gaseous active iodine.

The molecular sieve is a synthetic zeolite (18) with a definite crystalline structure containing a large number of small caves interconnected by a number of

Table IV. Yield and Contamination Data for Cadmium Separation Methods

Contaminating Ion	Reinecke Salt % contamination at pH 2.2	Reinecke Salt % contamination other 1N HCl	HPBZ % contamination at optimum conditions
Zn	0.1	0.25	8.0
Cr	0.12	0.20	4.0
Cs	1.0	0.25	10.7
Co	0.08	0.06	80+
Se	80+	-	5.5
Sb	80+	5.0	11.0
Ce Pr	0.05	0.003	5.0
Ir	0.10	0.10	9.0
Ru Rh	4.0	80+	80+
Sn	80+	0.07	5.2
Ag	80+	-	3.8
Tl	80+	-	4.5
Cd	69.8	55.3	80.0

still smaller holes or pores. These cavities and pores are precisely uniform in size. Normally these cavities contain water but the water can be driven off by heating the sieve, leaving the physical structure unchanged---with the empty cavities comprising almost 50% of the total volume of the crystals. In the absence of water any material can be adsorbed in the cavity if it can pass through the pores. Molecular sieves are available with pore sizes of 3, 4, or 5 Å, making possible separation of materials on the basis of molecular size. Polar molecules are generally most strongly adsorbed. These properties of the molecular sieve have been utilized in a number of molecular separations in gas as well as liquid phases.

The I^{131} tracer (19) used in these experiments was oxidized (20) in aqueous solutions along with a small amount of added carrier and distilled into an air stream drawn through a column containing the molecular sieve and finally through a sodium thiosulfate scrubbing system. The loss of tracer by adsorption on the walls of the equipment was found to be greatly reduced by application of "Dri Film" (21).

Preliminary experiments indicated a pronounced dependence of iodine adsorption on pore size. With the 3 Å form a small amount of iodine adsorbed which could be essentially quantitatively displaced by water vapor. This adsorption of iodine did not occur on a similar

column treated with Dri Film prior to the test and is assumed to be surface adsorption. With the 5 Å form, both iodine and water were readily adsorbed at the same time and retained on the column.

A measured decontamination of greater than 10^3 was obtained on a laboratory scale experiment with air pulled at ~50 cc per minute through a small column 8" by 0.5" containing 30 grams of type 5 Å adsorbent. Decontamination was also measured with a scaled up column 22" by 1.7" at an air flow of about 1 cfm. The distribution of activity along the length of this column obtained by measuring representative sections is shown in Fig. 9 to be around 10^5. The gas scrubbing system at the end of the column was used to confirm the decontamination factor and to prevent discharge of any active iodine to the open hood.

Essentially irreversible adsorption of iodine was found to occur in the presence of nitrogen oxides. A small column maintained under these conditions at 375°F, a temperature at which neither water or iodine will normally adsorb, gave an iodine decontamination of greater than 100 over a several hour period. The useful life of the column under these more rigorous conditions is not known.

The laboratory data indicate two promising possibilities for scaled up systems. A system of the type described in the previous paragraph could be used or a

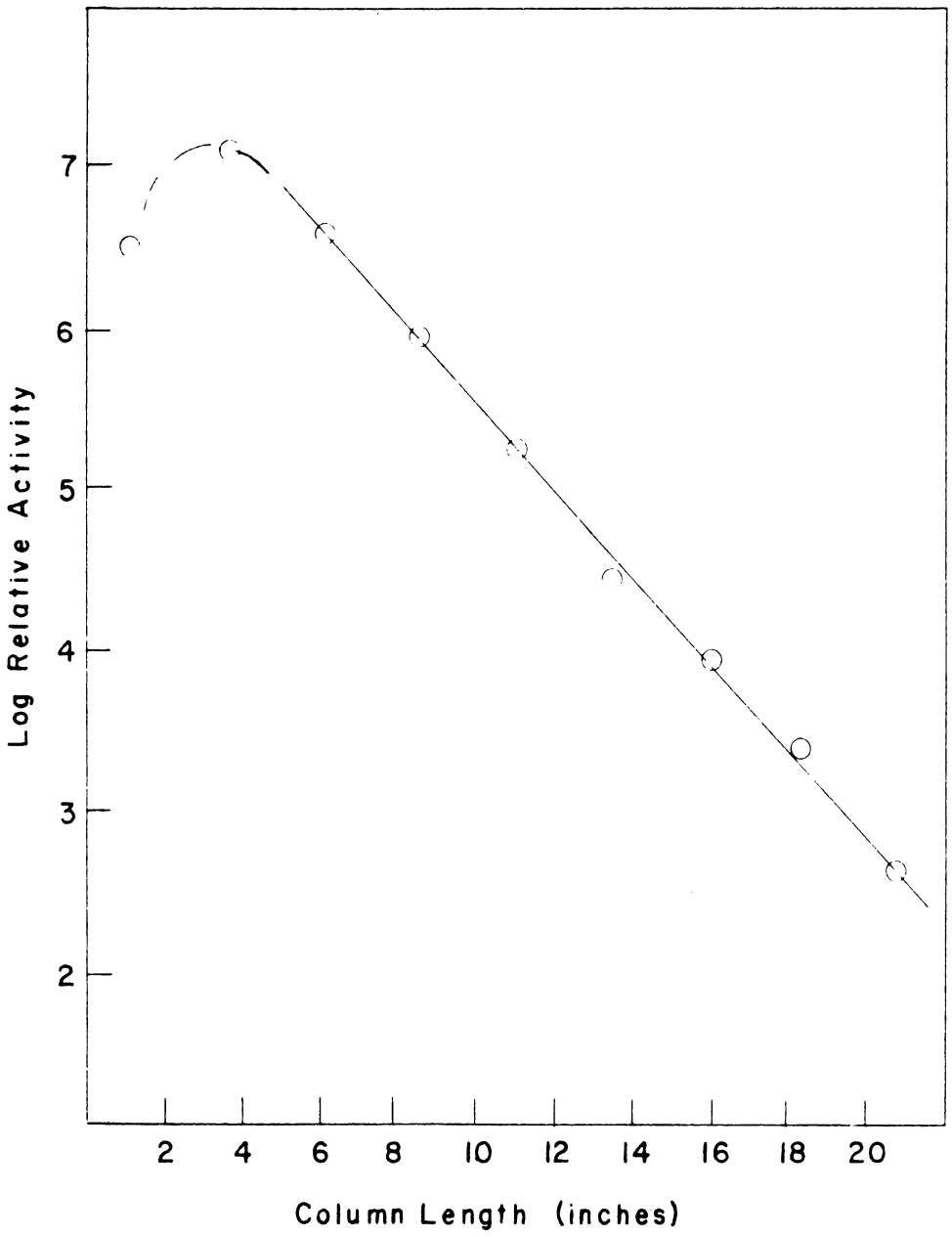


Figure 9. Distribution of I^{131} Adsorbed on 5A Molecular Sieve Column From Air Passed Through at 1 cfm.

system set up in which the air is dried by passing through one of a pair of 3 Å columns before passing through the 5 Å column. The drying columns would be alternately regenerated as necessary, simply by heating and purging. If the 5 Å column became saturated with iodine, storage for several months would allow quantitative decay of the iodine to inactive and desorbing xenon. This work has been described in a note to NUCLEONICS (22). (M. Wahlgren, W. W. Meinke)

C. Activation Analysis

Little work has been done with the portable neutron sources during the past year. Instead we have concentrated on preparation for use of the Michigan Reactor. With the reactor out of operation until very recently, however, it has not been possible to obtain experimental verification for the experimental plans described earlier (1).

1. Neutron Cross Section Graphs

Several years ago a plot was made of the atomic cross section for thermal neutron activation versus the half-life of the daughter product as a useful way of indicating and correlating nuclear reaction properties. The original graphs were based on the early compilation of cross sections by Hughes (23). When in 1955 Hughes and coworkers revised their cross section values (24) it was felt of interest to replot this graph and include the error limits as brackets on the points. This has been done and the results are shown in Figs. 10 and 11. These graphs have appeared in ANALYTICAL CHEMISTRY (25).

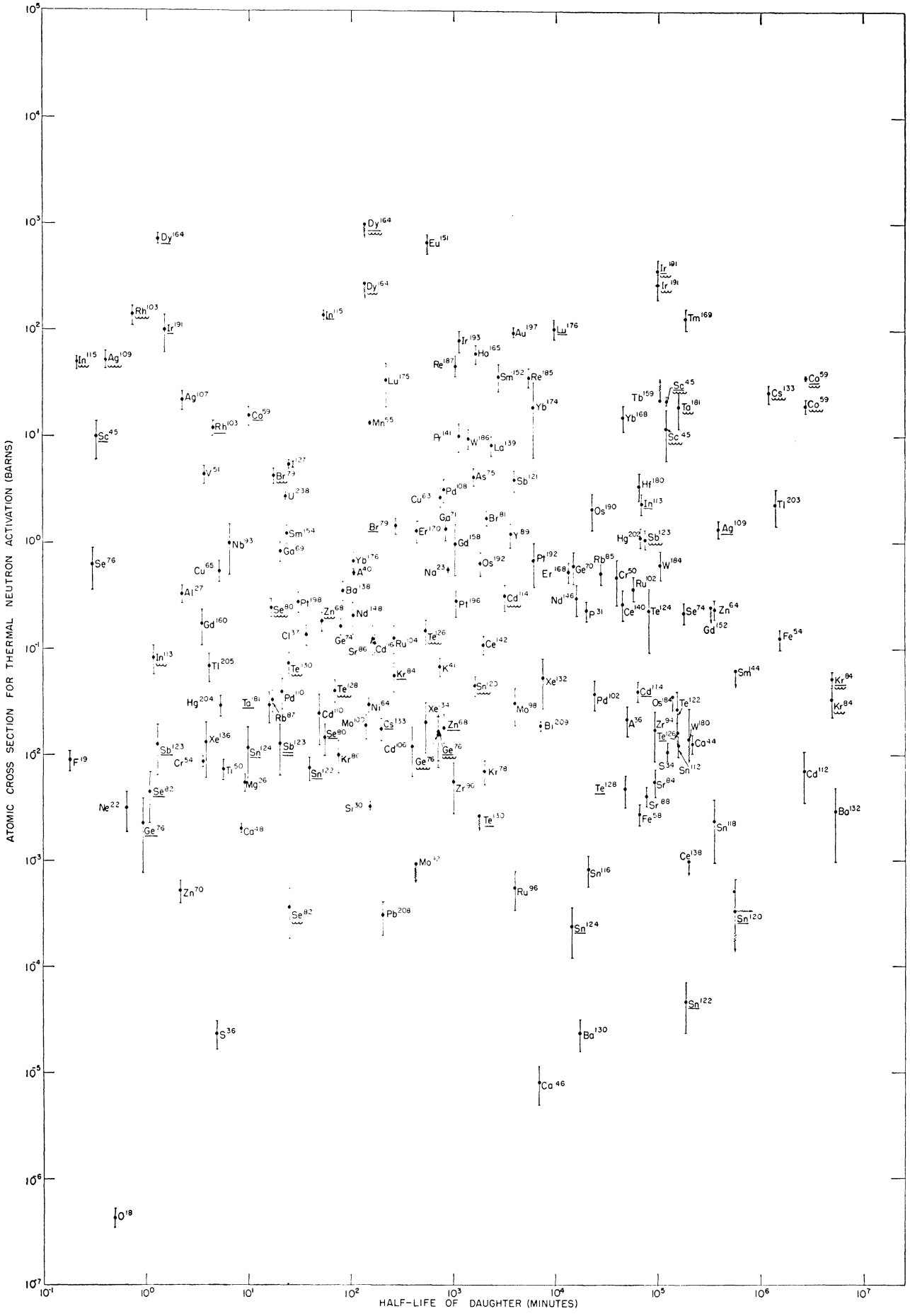


Figure 10. Atomic Cross Section For Thermal Neutron Activation of Isotopes Found in Nature vs. Half Life of Daughter Radioisotope Produced.

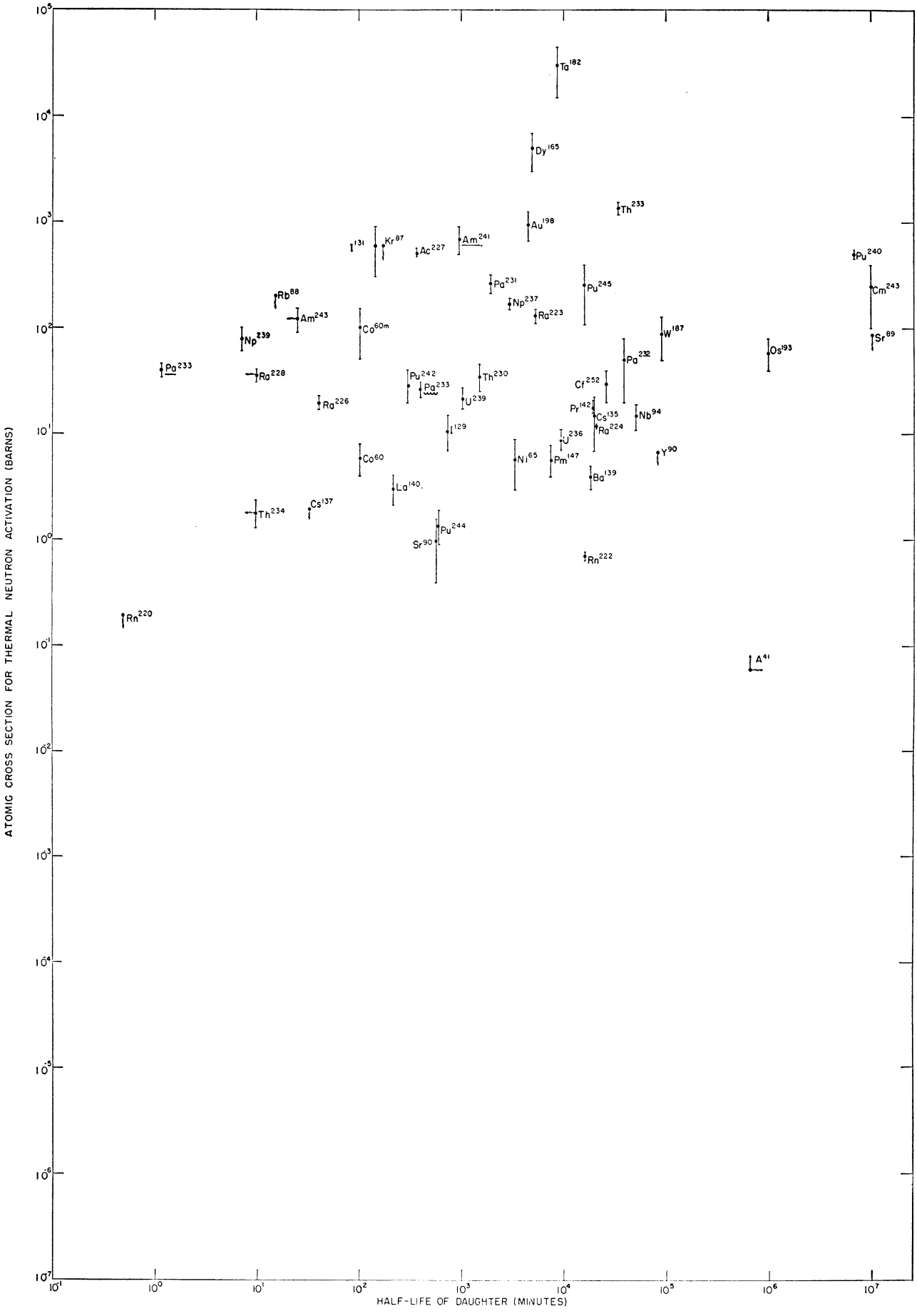


Figure 11. Atomic Cross Section for Thermal Neutron Activation of Isotopes Not Found in Nature vs. Half Life of Daughter Radioisotope Produced.

Figure 10 includes cross sections for all stable isotopes for which natural abundances are known. Figure 11 includes all cross sections given by Hughes for isotopes which are in themselves radioactive and which do not occur in nature. The atomic and isotopic cross sections are identical in Fig. 11.

An isotope that is underlined on the graph indicates that neutron activation produces a metastable daughter activity. A wavy line, on the other hand, indicates activation to the ground state of an isomeric pair. (Antimony-123 gives two metastable antimony-124 states and therefore has two underlines.) In most cases the metastable state decays independently by beta emission or contributes only a small amount to the activity of the ground state. In some cases, however, the metastable state decays completely into the ground state, thus augmenting its activity. The points with an underline and a wavy line are examples of this latter case and represent the total cross-section values for the formation of the ground state both directly and by decay of the short-lived metastable state.

As an example, stable cobalt-59 is activated to 10.4-minute cobalt-60m with a 16 ± 3-barn cross section and at the same time to the 5.28-year cobalt-60 ground state with a 20 ± 3-barn cross section. Since all the 10.4-minute metastable state of cobalt-60 decays directly to the ground state, the 5.28-year activity is produced with an effective cross section of 36.0 ± 1.5 barns.

These graphs contain all the (n,γ) cross-section values given by Hughes for thermal neutrons except for a few reactions where the cross sections are too small or the half lives too large to be plotted on the graphs. These values are given in Tables V and VI.

From these graphs it is possible to tell at a glance which elements will be activated with high sensitivity at different irradiation times. Furthermore, the graphs should prove helpful in estimating the activities of one isotope relative to another for specific conditions of irradiation.

It is interesting to note how poorly the absolute cross sections for most of the isotopes are known. Values for a few isotopes such as manganese-55 have been determined quite accurately. However, most of the cross sections for the remaining isotopes are known only to within 10, 20, or even 40%. Thus, it is of utmost importance when performing analyses by activation to utilize the standard analytical practice of comparison against a standard. This practice eliminates the necessity for knowing accurate cross-section values as well as absolute values for the geometry, counting efficiency, and the like of the experimental arrangement. Whenever absolute values of the cross section must be used, it can be expected that the work will be considerably less accurate. (W. W. Meinke and R. S. Maddock)

Table V. Thermal Neutron Cross Section Values Not Included in Figure 10

Parent	Log Half-Life of Daughter (minutes)	Log Atomic Cross Section for Thermal Neutron Activation (barns)	Standard Error of Cross Section (percentage $\pm$)
H^2	6.81	$\bar{8}.93$	1.8
Li^7	$\bar{2}.15$	$\bar{2}.48$	15.
Be^9	12.15	$\bar{3}.95$	10.
B^{11}	$\bar{4}.70$	$<\bar{4}.60$	--
C^{13}	9.46	$\bar{7}.00$	40.
N^{15}	$\bar{1}.08$	$\bar{8}.94$	33.
O^{18}	$\bar{1}.68$	$\bar{7}.62$	19.
Cl^{35}	11.20	1.34	67.
A^{38}	8.11	$\bar{4}.70$	25.
K^{39}	14.83	0.45	67.
U^{234}	14.57	$\bar{3}.61$	14.
U^{235}	13.11	$\bar{1}.88$	4.7

Table VI. Thermal Neutron Cross Section Values Not Included in Figure 11

Parent	Log Half-Life of Daughter (minutes)	Log Atomic Cross Section for Thermal Neutron Activation (barns)	Standard Error of Cross Section (percentage ±)
Th^{228}	9.58	2.09	12.
U^{232}	10.93	2.48	67.
U^{233}	11.11	1.72	3.8
Pu^{238}	10.11	2.63	16.
Pu^{239}	9.54	2.50	5.1
Pu^{241}	11.41	2.58	13.
Pu^{243}	<8.72	2.00	50.
$\underset{\sim}{Am}^{241}$	8.41	<1.70	--
$\underline{Am}^{241}$	8.41	<2.28	--
Cm^{242}	7.72	1.30	50.
Cm^{244}	8.41	1.18	67.
Cm^{245}	9.00	2.30	33.

2. Activation Analysis with the Michigan Reactor

It has not been possible to date to begin the program outlined in the last progress report (1) with the Michigan Reactor. The plans, however, remain the same--to explore the uses of short half-lived radioisotopes in activation analysis. The advantages of utilizing short half-lived isotopes are many. It can be seen vividly for example from Fig. 10 that activation analysis for silver can be made using either the 2.3-minute silver-108 from silver-107 or the 270-day silver-110 from silver-109. If the shorter half-lived material is used not only is the cross section greater by a factor of ~20 but it is possible to bombard to saturation in a matter of minutes whereas you can only approach a small fraction of saturation of the silver-110 in a few days bombardment. A number of other examples of this can be seen from the graph.

In addition the utilization of short half-lived materials in activation analysis can often allow the completion of the irradiation and analysis within fractions of an hour rather than spreading it over several days as in the standard present day techniques.

One of the limitations of this approach, however, is the fact that rapid chemical separations must be made on the sample. In the case of silver this is made possible by utilizing the isotopic exchange technique developed in this laboratory. Thus if a sample can be put into solution rapidly it should be possible to analyze for silver in a matter of 10 minutes or so. If this rapid solution is not

possible it may be necessary to include some pretreatment of the sample to get it into a soluble form. Any pretreatment, however must be done with the appreciation that many of the advantages of activation analysis may be invalidated by the possible introduction of trace amounts of the desired element in the reagents.

Among the elements that are being studied in connection with this program are vanadium with its 3.8-minute vanadium-52 daughter, rhodium with its 4.4-minute rhodium-104 daughter, and silver with its 2.3-minute silver-108 daughter. Rapid separations have been developed for these elements and tested with long-lived tracers. They will be further tested with the reactor at the earliest opportunity.

3. Utilization of Various Isotopes for Activation Analysis

The graphs of Figs. 10 and 11 are merely plots of cross section versus the half life and do not in any way indicate the ease with which a particular isotope can be measured. Indeed certain isotopes such as dysprosium-164 given on that chart decay in such a fashion that it would be very difficult to detect them with very high sensitivity (26). Therefore it is essential in planning activation analyses to have an appreciation for the decay schemes of the different isotopes which might be formed.

Any such evaluation must, of necessity, make a number of standardizing assumptions. In the present work these assumptions have been that counts are taken whenever possible with an end-window Geiger counter to obtain maximum sensitivities and that the Geiger counter has a

fifteen per cent geometry. Self absorption corrections have also been included. Whenever decay schemes are such that only a small percentage of the isotope decays by beta emmision, use of scintillation well counters was assumed.

Data from these evaluations have been incorporated into a table, which has been submitted along with a review article on activation analysis, to ANGEWANDTE CHEMIE. It is planned to issue this table in the form of an AEC report, which can be made available to interested parties and possibly also to plot these calculated sensitivities versus half life in a graph similar to Fig. 10 for easy reference.

It is realized that in some cases interpretation of the decay scheme may be open to question but at least this work is an attempt to evaluate the measurement efficiencies and hence the practical sensitivities of different isotopes formed by thermal neutron capture. (U. Schindewolf)

4. Fundamental Review of Nucleonics in Analytical Chemistry

Again this year the author is making a review of the literature of fundamental developments in nucleonics that are applicable to analytical chemistry. Since most developments in nucleonics are applicable to analysis this review becomes quite all-inclusive. The previous review made two years ago included 593 references worthy of note without covering many specific references on instrumentation. At the present time some two thousand or more references have been found in the literature of the past two years; these

will probably be condensed to about one thousand references for the article. The deadline for submission of the article is December 1, 1957, and it will appear in the April, 1958 review issue of ANALYTICAL CHEMISTRY. (W. W. Meinke)

IV PERSONNEL, PUBLICATIONS, TALKS

A. Personnel Listing

Staff Meinke, W. W.

Post Doctoral Bruninx, E. (Michigan Memorial Phoenix Project No. 121)

Fourage, J. (Exchange student from Belgium)

Schindewolf, U.

Graduate Students Anders, O.*(')

DeVoe, J.*

Gardner, D.*(') (Eastman Kodak Fellowship, 1956-57)

Netzel, D.(') (Summer only)

Wahlgren, M.*

Undergraduate Students Holbrook, S.**(')

Grindahl, G.**(')

Sargent, M.**

Non Staff Maddock, R. S.

Typing Blackburn, J.**

Greenley, B.**(')

Schwing, J.**

Showerman, S.**(')

Electronics Shideler, R. W.*

* Half time

** Hourly

(') Terminated

B. Papers and Reports Published

1. High Energy Excitation Functions in the Heavy Region. W. W. Meinke, G. C. Wick and G. T. Seaborg. J. Inorg. and Nucl. Chem. 3, No. 2, 69 (1956). 23 pages, 11 figs.
2. Excitation Functions and Cross Sections. Compiled by O. U. Anders and W. W. Meinke. Document number 4999, American Documentation Institute, Library of Congress, Washington, D. C. (Revised June, 1956).
3. A Simplified Photographic Coincidence Method. Donald G. Gardner and Ronald W. Shideler. U. S. Atomic Energy Commission Unclassified Report AECU-3397 (April 1957). 6 pages, 3 figs.
4. Absolute (d, α) Reaction Cross Sections and Excitation Functions. Oswald U. Anders. U. S. Atomic Energy Commission Unclassified Report AECU- . PhD Thesis, April 26, 1957. 251 pages, 71 figs.
5. Nuclear Decay Scheme Analysis and Characterization Studies of (d, alpha) Reaction Products. Donald Glenn Gardner. U. S. Atomic Energy Commission Unclassified Report AECU- . PhD Thesis, May 15, 1957. 232 pages, 67 figs.
6. Fast Geiger Mueller Counters. A Literature Review to May 1947. W. Wayne Meinke.
7. Neutron Activation Cross Section Graphs. W. Wayne Meinke and R. S. Maddock. Anal. Chem. 29, 1171 (1957). 4 pages, 2 figs.
8. Molecular Sieves Adsorb Iodine-131 From Air. Morris A. Wahlgren and W. Wayne Meinke. Nucleonics 15, No. 9, 156 (1957). 2 pages, 4 figs.

9. Decay Scheme of Co^{62}. Donald G. Gardner and W. Wayne Meinke. Phys. Rev. (In Press).
10. Evaluation of Radiochemical Separation Procedures. Duane N. Sunderman and W. Wayne Meinke. Anal. Chem. (In Press). (A combination of 4 papers--"Radiochemical Separations I: Barium Strontium and Calcium", "Radiochemical Separations II: Separation of Radioactive Silver by Isotopic Exchange "Radiochemical Separations III: Silver" and "Iron and Lanthanum Hydroxides as Scavengers in Radiochemical Separations")
11. New Determination of the Phosphorus-32 Half-Life. O. U. Anders and W. Wayne Meinke. Nucleonics (In Press).
12. Revised Decay Scheme of Y^{92}. Donald G. Gardner and W. Wayne Meinke. Phys. Rev. (Submitted).
13. Beta-Ray Spectroscopy Using A Hollow Plastic Scintillator. Donald G. Gardner and W. Wayne Meinke. Nucleonics (Submitted).
14. Nuclear Technique Training For Analysts Lagging. W. W. Meinke, W. H. Beamer and D. C. Stewart. Anal. Chem. 29, 19A (1957). 5 pages, 3 figs.

C. Talks

1. O. U. Anders, "Absolute Yields of Deuteron Induced Nuclear Reactions with Emphasis on the (d, α) Reaction", U. of M. Chemistry Department Colloquium, Ann Arbor, February 21, 1957.
2. D. G. Gardner, "Nuclear Decay Scheme Analysis", U. of M. Chemistry Department Colloquium, Ann Arbor, February 28, 1957.
3. W. W. Meinke, "Nuclear Chemistry and the Michigan Reactor", Student Affiliate of American Chemical Society, Ann Arbor, March 27, 1957.
4. W. W. Meinke (with D. N. Sunderman), "Radiochemical Separations of Silver", Analytical Division, American Chemical Society, Miami, Florida, April 10, 1957.
5. D. G. Gardner (with W. W. Meinke), "Beta-Ray Spectroscopy Using a Hollow Plastic Scintillator", Industrial Nuclear Technology Conference, Chicago, Illinois, May 15, 1957.
6. W. W. Meinke, "Radiochemical Training and the University", Chairman of Panel on Education and Training in Nucleonics for Analysis, 10th Annual Summer Analytical Symposium, Purdue University, Lafayette, Indianna, June 15, 1957.

V ACKNOWLEDGEMENTS

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The help of the Isotopes Division of the Atomic Energy Commission in supplying both enriched and radioactive isotopes for use in this work is acknowledged.

We also appreciate the assistance of the Michigan Phoenix Memorial Project in generously supporting part of this program of research under Phoenix Projects Nos. 95 and 121, and in making available additional laboratory space for this research in the Phoenix Building.

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2. Progress Report 4, Nuclear Chemical Research and Radiochemical Separations, University of Michigan, Project No. 7, Contract No. AT(11-1)-70, U. S. AEC. W. W. Meinke Project Director. U. S. Atomic Energy Commission Report AECU-3116 (November 1, 1955).
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