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Contract Number AT-33-1-GEN-53

MOUND LABORATORY

Operated By

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MIAMISBURG, OHIO

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NEUTRON SOURCE PROGRESS REPORT

Date: October 1-31, 1948

Distributed: DEC 9 1948

Prepared by: J. L. Richmond

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NEUTRON SOURCE GROUP

L. L. Bentz, J. H. Birden, M. R. Hertz, J. L. Richmond, W. J. Schrantz, and R. M. Watrous

<u>General Problem</u>	<u>Specific Problem</u>	<u>Man-Months</u>	<u>Job Classification</u>
All Problems (Group Leader)		1	202
I. Study of Production Methods	A. Postum-Beryllium Neutron Sources	1 1/2	205-C
	B. Postum-Fluoborate Neutron Sources	1	205-C
	C. Postum-Miscellaneous Neutron Sources	1	205-C
II. Engineering Study of Dry Boxing and Shielding	Carried out in Con-junction with above Problems	1	206-C
III. Neutron Counting	Assembly of Equipment	1/2	205-C

ABSTRACT

I-A. Postum-Beryllium Neutron Sources

Three small postum-beryllium sources were prepared and sent to Unit IV for neutron standards.

A 15 millicurie source was made and shipped to Brookhaven National Laboratory.

Beryllium sols prepared by arcing in methyl alcohol had particles in the range of 500 Å. They could be coagulated by applying D.C. voltage, but were difficult to filter. Two neutron sources were prepared having 62 and 14 millicuries of postum with efficiencies of 15 and 41 per cent, respectively. It is probable that the amount of beryllium was such that a thick target was not obtained, causing the low efficiencies.

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A Report of the Work Done in Preparing Neutron Sources from Polonium and Beryllium between September 5, 1945 and May 25, 1947, MLM-187, by John H. Birden was distributed.

I-B. Postum-Fluoborate Neutron Sources

An unsuccessful attempt was made to prepare a postum-sodium fluoborate-sodium fluoberyllate source in which postum was added in hydrochloric acid and a small amount of hydrofluoric acid added to convert the chloride to fluoride. Apparently, the chloride loses water of crystallization at elevated temperature, causing a pressure which forces out the contents of the container.

When hydrofluoric acid alone was used as solvent for the postum, the source was prepared without difficulty. Source PB-123 contained 39.8 cases of postum on January 19, 1948. The neutron count varied with successive periods of heating, increasing from 8.6×10^6 n./sec. to 1.07×10^7 n./sec., then decreasing to 9.8×10^6 n./sec.

I-C. Postum-Miscellaneous Neutron Sources

Deplating postum in sulfur dioxide water and adding to sodium sulfide gave a source of 72 per cent efficiency. Postum was not dissolved by carbon disulfide.

Hydrogen sulfide water will remove postum from platinum but holds it in suspension instead of in true solution. When such a suspension was added to sodium sulfide, the efficiency of the source was less than when the postum was put in solution by the addition of hydrochloric acid.

Deplating postum in a water solution of sodium sulfide and evaporating the water gave a source of 66.8 per cent efficiency. A microfoil with 0.1 case of postum was immersed in carbon tetrachloride. The neutron count increased for four days until an efficiency of 65 per cent was reached. On removal of the microfoil, on which only 10 millicuries of postum remained, the efficiency dropped to 51 per cent.

A postum-sulfur monochloride source prepared by placing a microfoil in the liquid had an initial efficiency of 69 per cent, increased to 79 per cent and leveled off at 67 per cent.

A postum-antimony pentafluoride source prepared by placing a microfoil

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in the liquid had an initial efficiency of about 75 per cent and remained near that figure.

II. Engineering Study of Dry Boxing and Shielding

Nothing to report, except as reported under other headings.

III. Neutron Counting

Procurement of equipment for neutron counting at Mound Laboratory has been initiated.

A seminar was given October 14, 1948 in which possible methods of neutron counting were outlined. It is planned to measure the reaction product from an alpha, neutron reaction; for example, carbon from alpha on beryllium. This may be attempted spectrographically and chemically.

Another method which may be of value is the reaction of alpha on a radioactive isotope, one atom of the radioactive material being converted for each neutron produced. In this case a different count on the radioactive material would be a measure of the neutrons.

DETAILED REPORT

I. Study of Production Methods

A. Postum-Beryllium Neutron Sources

Three sources (WS-9, WS-10, and WS-11) have been prepared and shipped on October 25, 1948 to Unit IV. Source WS-9 was made from sintered beryllium rod 1/16" in diameter and 1/8" in length. Two pieces of this rod were necessary to obtain the desired flux, probably because the active solution was not pure.

Sources WS-10 and WS-11 were made by placing sintered beryllium rods 1/8" x 1/8" in the tip of 4" lengths of Tygon tubing and pulling active solution over the beryllium by means of a pipette. Postum was volatilized from platinum into quartz and taken up in 1.5 N nitric acid, as postum dissolved directly from platinum did not scrub on beryllium. The sources were rinsed with methyl alcohol and dried. They were placed at the centers of brass cylinders 1/2" x 1/2", which were then nickel coated.

The sources were counted three times to check the neutron decay with postum decay, as shown in Table I.

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Table I

NEUTRON COUNTS

Source No.	Neutron/sec. 10/15/48	Neutrons/sec. 10/20/48		Neutrons/sec. 10/25/48	
		By Count	By Decay	By Count	By Decay
WS-9	3.22×10^3	3.05×10^3	3.16×10^3	2.91×10^3	2.97×10^3
WS-10	9.77×10^3	9.52×10^3	9.58×10^3	9.31×10^3	9.29×10^3
WS-11		2.98×10^4		2.87×10^4	2.91×10^4

A postum-beryllium source (PB-129) of 15 millicuries on October 25, 1948 was sent to Brookhaven National Laboratory on the same date. The emission was 2.78×10^4 n./sec. The method of preparation and the container construction were the same as for source WS-11.

A number of runs were made on the arc apparatus to produce sufficient beryllium sol for various tests. Performance of the apparatus was similar to that in previously reported results.¹

Samples of the original beryllium powder, the sintered beryllium electrodes, and the powder obtained by evaporating the sol were submitted to Unit I for spectrographic analysis. The only change in analysis through the process was the gain of 0.5 - 1.0 per cent silica, apparently from the quartz tube in which the electrodes are sintered. A sample of the sol was also submitted for an estimate of particle size by use of the electron microscope. The particle size is apparently in a fairly narrow range at about 500 Å diameter. The electron diffraction pattern obtained showed definitely that the particles are crystalline in nature.

When 90 - 225 volts D.C. was placed across platinum electrodes immersed in the sol, coagulation in a loose floc occurred at the anode. This floc could be centrifuged; but upon attempting to filter, it either broke up and went on through or broke up and plugged the filter. Small filter cans were machined from 4 mm. sintered beryllium rods. Cans from 200 - 230 mesh beryllium powder were the most nearly successful. After a

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short time the filters became almost plugged and from this point on the floc was filtered out at a very slow rate.

Source H-1 was made by evaporating coagulated beryllium sol containing postum into a quartz capillary. About 200 millicuries of postum was electroplated on a platinum electrode, after which the electrodes were immersed in the sol. Ninety volts D.C. was placed across the electrodes with the one on which the postum had been plated as the anode. Thus, an attempt was made to deplete the postum at the same time beryllium was being coagulated on the same electrode. About 100 mc. was depleted by this method. The tube was then centrifuged to consolidate the floc in the capillary section, then evaporated. The capillary section of the tube was cut off and counted. The neutron flux was 2.772×10^4 n./sec. while the calorimeter value was 61.6 mc., corresponding to an efficiency of 15.5 per cent. The low efficiency was believed due to the fact that the beryllium powder (approximately 1.4 mg.) spread over the surface of the capillary would only have an average thickness of $1-2 \mu$, considerably less than that for a thick target.

Source H-2 was made by similar methods, except that rather than evaporating, an attempt was made to filter the beryllium floc into the bottom of a small sintered beryllium can. Only a small portion of the total beryllium (1.4 mg.) could be filtered before the filter can plugged. The flux of the source (H-2) was 1.654×10^4 n./sec. and the calorimeter value 14.2 mc. corresponding to an efficiency of 40.9 per cent. It is believed that if filtering could be accomplished successfully a higher neutron efficiency could be obtained.

In an attempt to produce a more easily filterable floc, solutions were made in benzene and diisopropyl ether. No increase in filterability was observed.

I-B. Postum-Fluoborate Neutron Sources

Work was continued on preparation of the thirty-five case mock fission neutron source that had been begun in September. The second preparation was made in which the postum was deposited as the chloride, target powder was added, several separate portions of 24 per cent hydrofluoric acid solution were added and evaporated. The hydrofluoric acid was added to distribute the activity throughout the target powder and to effect a change of postum chloride to postum fluoride. The powder was dried, tamped and the source cutoff. After the cutoff, the red ooze came from the can again and the source had to be dissolved.

A third attempt was made to prepare the source. A change in tactics was indicated, since it was evident that the postum-chloride could not be

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changed to the fluoride and it was the chloride that was causing the difficulties. A new top was made for the evaporation apparatus with everything being the same except that Lucite was substituted for glass in making the window (Figure 1) since glass would be etched by hydrofluoric acid. A small amount of the total target powder was added to the evaporation can and 250 lambda of 24 per cent hydrofluoric acid added to wet the powder. The acid was evaporated and 250 lambda of hydrofluoric acid containing postum added to the powder through the use of a polystyrene pipette. This solution was evaporated. Additions and evaporations were made until all of the postum was deposited on the powder. The remainder of the target powder was added and several separate amounts of hydrofluoric acid added and evaporated to distribute the postum. The powder was dried for several hours and then tamped into a pellet. The source and can were cutoff with a tubing cutter and the can coated with five mils of nickel. The nickel coated can was placed in a brass container and was capped with a screw lid made of brass. This assembly was coated with five mils of nickel. The source furnished enough heat to coat itself with nickel in both coatings. The source was calorimetered and neutron counted. After counting, the source was placed in a complete vacuum and allowed to heat itself for one-half hour. It was found to have increased in efficiency after this operation so the operation was repeated until the source leveled off in efficiency.

The source was shipped to Los Alamos October 21, 1948.

Table II

DATE ON NEUTRON SOURCE PB-123 CALORIMETER VALUE 39.82 10/19/48

<u>Neutron* Counts</u>	<u>Compared To Stand. #49</u>	<u>Compared To Stand. Al-1</u>	<u>Date</u>	<u>Efficiency</u>
1	6.885×10^6 n./sec.	8.615×10^6 n./sec.	10/13/48	49.3%
2	7.672×10^6	9.757×10^6	10/14/48	56.1%
3	8.333×10^6	1.066×10^7	10/15/48 A.M.	61.6%
4	8.25×10^6	1.026×10^7	10/15/48 P.M.	59.3%
5	7.656×10^6	9.765×10^6	10/19/48	57.6%

* Neutron count number one is original count. All others are taken after one-half of self-heating before each count.

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—FIGURE 1—

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APPARATUS USED IN PREPARING NEUTRON
SOURCE PB123

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III. Postum-Miscellaneous Neutron Sources

A postum sodium sulfide neutron source, LB-53, 0.130 c. October 5, 1948, had an efficiency of 72.0 per cent. The source was prepared by deplating postum at 2.4 milliamps (current density - 2 milliamps./sq. cm.) into a one ml. solution of freshly gassed sulfur dioxide water. The above solution, with the deplated activity, was transferred to 0.2051 gm. of sodium sulfide in a platinum cylinder. The liquid was slowly evaporated by means of an electric heater, heated at 150°C. - 160°C. and prepared for neutron counting. A slide test at the time the activity was transferred to the target material indicated approximately 0.020 c. in solution. From the above calorimeter value of the source 0.130 c. and the 0.020 c. in solution at the time of transfer, it was indicated that approximately 85 per cent of the activity had been in suspension.

An experiment was conducted on the solubility of postum in carbon disulfide. A platinum foil with postum was inserted into one ml. of the solution. After approximately two hours a slide count on the solution indicated that less than 0.1 millicurie had gone into solution. Due to evaporation losses additional amounts of carbon disulfide had to be added to maintain the level at one milliliter. After four days, less than 0.3 millicurie had gone into solution. From this data it would appear that postum is insoluble in carbon disulfide.

COMPARISON OF SOURCES LB-51 WITH LB-52

<u>Source</u>	<u>Cases</u>	<u>Type Source</u>	<u>Q-Solvent</u>	<u>Efficiency</u>
LB-51	0.204 c.	Q-Na ₂ S	H ₂ S Water	89.4%
LB-52	0.145 c.	Q-Na ₂ S	.1 N HCl	103.0

It can be seen that the efficiency is higher in the case where the activity was dissolved in hydrochloric acid. It was thought the efficiency in this particular source might be lower due to the formation of impurities with lower neutron fluxes. Since the efficiency of LB-52 was nearly 14 per cent higher than LB-51, it was thought that if one ml. of .1 N hydrochloric acid was allowed to digest the LB-51 source for several days at room temperature, the activity would be redissolved. Then with the vaporizing of the solution it was hoped to have a more homogenous deposit of postum on the surface of the sodium sulfide. From previous results of dissolving postum in sulfur dioxide water and hydrogen sulfide water and then transferring the activity onto the

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target material, it was found that approximately 15 - 20 per cent of the activity was in solution and the remainder apparently was in suspension. This could account for the decreased efficiency of LB-51. The source in question was allowed to digest with one milliliter .1 N hydrochloric acid for seven days and then it was dried and neutron counted. The efficiency before and after treatment was 70.2 per cent and 69.2 per cent, respectively. The decrease in efficiency from the date the source was made cannot be explained.

A postum-sodium sulfide source, LB-54, 0.107 c. October 7, 1948, had an efficiency of 66.8 per cent. The source was prepared by deplating postum in a one ml. water solution of 0.2034 gm. of sodium sulfide. After deplating three hours the source was heated to evaporate to dryness in the usual way.

Postum Liquid Neutron Sources

A postum-carbon tetrachloride neutron source, LB-55, 0.108 c. October 11, 1948, had a top efficiency of 65.1 per cent. The source was prepared by adding a microfoil with postum to a brass cylinder nearly filled with carbon tetrachloride. At the time the source was prepared, the efficiency was 49 per cent. For two days the efficiency increased, then dropped off nearly six per cent on the third day and on the fourth day it reached its top efficiency. The efficiency remained the same the fifth day. Thereupon the foil with the activity was removed from the source and the efficiency dropped to 51 per cent. Another calorimeter value of the source indicated that only 0.010 c. had been removed with the foil. Work is in progress to determine the reason for decreased efficiency.

A postum-sulfur monochloride source, LB-56, 0.185 c. October 15, 1948, had a top efficiency of 78.8 per cent. The source was prepared by adding a microfoil with postum to a stainless steel cylinder nearly filled with sulfur monochloride. The efficiency was 69 per cent at the time the source was prepared and steadily rose until it reached its top efficiency (78.8 per cent) after the second day. After dropping to 67.5 per cent the third day, the source was uncapped to see if the liquid had evaporated or leaked out. The liquid level was approximately the same as when the source was prepared. The efficiency increased to 71.9 per cent on the fourth day and dropped back on the fifth day to 67.1 per cent.

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A postum-antimony pentafluoride source, 0.215 c. October 14, 1948, had a top efficiency of 75.8 per cent at the time it was prepared. The source was prepared by adding a microfoil with postum to a stainless steel cylinder nearly filled with antimony pentafluoride. The neutron efficiency averaged around 74 per cent on being neutron counted on four different days.

II. Engineering Study of Dry Boxing and Shielding

See Abstract.

III. Neutron Counting

See Abstract.

FUTURE PLANS

See Section III of Abstract.

REFERENCE

1. Neutron Source Progress Report, MLM-178, August 1-31, 1948.

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