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MOUND LABORATORY

Operated By

MONSANTO CHEMICAL COMPANY

MIAMISBURG, OHIO

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REPORT FOR RESEARCH

ON

SUBSTITUTE MATERIALS

JULY 2, 1951 TO OCTOBER 1, 1951

(POLONIUM-208 VOLUME)

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Date: November 5, 1951

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SUMMARY

Mound Laboratory is cooperating on a joint development program directed toward the production of polonium-208 by proton irradiation of bismuth The Electromagnetic Division of Oak Ridge National Laboratory is operating a high-current cyclotron for the production of polonium-208 on an experimental scale. The California Research and Development Corporation is constructing the Mark I, linear accelerator at Livermore, California, which, at some unspecified future date, is planned to be converted from its original deuteron beam to a proton beam for the production of polonium-208. During this quarter, the accelerator-target-design problem has been transferred from the California Research Corporation, which now is no longer participating in the program to the California Research and Development Corporation. Mound Laboratory will process the polonium produced by any of these machines

At the present time Mound Laboratory is evaluating and processing the experimental targets produced at Oak Ridge. Also special problems are being studied for the other participating organizations because of the experience at Mound Laboratory in handling polonium at all levels.

During the past nine months the amount of polonium-208 received from Oak Ridge has increased from 14 microcuries to 57 millicuries to 992 millicuries. The larger amounts of polonium have made it possible to purify the polonium without the use of carriers However, the levels are still sufficiently low that considerable research was necessary to reduce the amount of material being recycled and increase the overall recovery.

Autoradiographs of the alpha and gamma activity on target surfaces have shown that a portion of the proton beam is being expended in the leading edge of the target: in the aluminum target backing, when holes are burned through the bismuth, and on small pieces of impurities in the bismuth. The extent of the loss of polonium production is not known because ORNL has recently found that the theoretical production calculated for a given target exposure was too high since beam energies were lower than preliminary calculations had indicated In general the successful efforts of ORNL to handle higher beam currents have not made it possible at the same time to reduce the relative gamma activity of the targets. This still remains a problem.

The average purity of the polonium which has been processed is 78.0 per cent polonium-208 by weight. A purity of 83.2 per cent was obtained by repeating the purification steps on one batch It seems probable that a large portion of the impurity must be polonium-209, which has a much greater half life The amount of polonium-209 calculated by the proton excitation curve is somewhat uncertain because of uncertainties in the beam energy, and pulse-height analyses involve the uncertainty in the half life of polonium-209, but the orders of magnitude corresponds with the amount of impurity found by mass assay (p. 6).

A mass spectrograph is being set up to establish the polonium-209 content. This study is being conducted at Mound Laboratory because the large quantity of alpha activity involved in the measurement constitutes a serious contamination problem (p. 44).

During the previous quarter a study was made of the behavior of vaporized polonium in a vacuum system in an effort to determine where the polonium lost from a target would eventually end up in a cylotron or linear accelerator This quarter a study has been made on the migration of polonium in a vacuum system in the absence of temperature gradients. It has been found that polonium migrates from a spot source in an evacuated chamber, and the degree

of migration increases with increasing temperature. (At atmospheric pressure very little migration occurs.) The migration evidently occurs in the process of evacuating the chamber and bring it back to atmospheric pressure, since the degree of migration was not found dependent upon the time held under vacuum. Autoradiographs of the contaminated surfaces showed particles of polonium, rather than an even film, which indicated that migration was not the result of polonium vapor (p.26).

Preliminary to the investigation of thin covering materials to prevent the loss of polonium from grazing-incidence targets in the ORNL cyclotrons, a study has been made of the loss from uncovered bismuth at 200°C. After 30 hours of heating, the rate of loss of polonium from bismuth containing 3.84 millicuries per gram levels off to about 1.8×10^{-6} millicurie per hour per square centimeter (p. 37)

Preliminary work has started on methods of decontaminating the copper cyclotron dees used in the production of polonium-208 (p.43).

Problem Title – **Purification of Polonium from ORNL Targets**

Report By – **G. D. Nelson**

Work Done By – **G. L. Fox, H. W. Kirby, and G. D. Nelson**

INTRODUCTION

Since the first of this year Mound Laboratory has been cooperating with Oak Ridge National Laboratory in a study of polonium-208. The polonium is produced by proton bombardment of bismuth in the 86-inch cyclotron at Oak Ridge, and the irradiated targets are sent to Mound for assay and recovery of polonium. The Electromagnetic Research Division of ORNL plans to prepare sufficient polonium so that Mound Laboratory can send one curie of purified material to Los Alamos about November 1, 1951.

Results of alpha and gamma assays of 25 targets have been reported previously. A survey of the gamma-active constituents in the bismuth and aluminum backing material of the targets was also presented.[1] Efforts to concentrate and prepare trace quantities of carrier-free polonium by the tellurium process were successful.[2] and it was hoped that, as the polonium inventory increased, material could be prepared by a stannous chloride reduction of the polonium carrier free followed by ammonia precipitation and electrolysis onto platinum as the purification steps.

This report presents results of assays on 28 targets received from July 1 through September 30, 1951, and discusses purification experiments and other associated studies made on ORNL polonium

DETAILED REPORT

Assay of Targets 35 - 69

When received, a target was assayed for alpha and gamma activity. The gamma count was compared with the count from an equilibrium radium standard in the same position as the target. An autoradiograph of the alpha activity on the surface of the target was then made. Starting with Target 59 a gamma measurement in terms of roentgens per hour and an autoradiograph of the gamma activity were also made. The amount of bismuth in a target was obtained by the difference in weight before and after the bismuth was dissolved. To dissolve the bismuth, the target was heated on a hot plate in a photographic developer tray with the front face immersed in concentrated nitric acid. The resulting solution of the bismuth containing the polonium was converted to the chloride and assayed for alpha activity by mounting an aliquot on a slide for a parallel-plate count.

The assay data are given for each target in Table I along with the surface dimensions, calculated alpha yield (determined by ORNL), the per cent of the estimated alpha yield achieved, and the ratio of gamma activity to alpha activity present. The ratio of gamma activity to 100 millicuries of alpha activity is plotted in Figure 1.

Discussion of Results of Target Assay

The size of the target has been increased from 2 by 6 inches to 6 by 6 inches, and one target 6 by 10 inches in area was received. This greater area allows for increased spread

of the beam.[3] In the autoradiographs shown in Figures 2 - 6 "N" indicated north, the direction from which the proton beam approaches the targets at grazing incidence.

The alpha yield has been as much as 42.7 millicuries on a 2 by 6 inch target, 153 millicuries on a 6 by 6 inch target and 181 millicuries on the one 6 by 10 inch target. The yields on Targets 59, 62, 64, 65, and 69 were all greater than 100 millicuries each and totalled 730 millicuries.

The average alpha yield was 61.3 per cent of the calculated yield. Since Table I was prepared, it has been learned that the calculated alpha-yield values should be reduced by perhaps as much as 45 per cent, since the beam energy has been reevaluated from 22 to 19.2 million electron volts.[4] The lower beam energy reduces the calculated polonium-208 yield, increases the relative polonium-209 yield, and raises considerably the per cent of the calculated alpha yield recovered during processing at Mound Laboratory (Table I)

Autoradiographs made of Targets 59 and 62 throw some light on the problem of target yields. The alpha yield was above average for Target 59. The gamma level was comparatively low apparently because the alpha activity was well distributed in the bismuth and because there was little surface melting. Considerable gamma radiation was produced at the north edge of the target and in impurities in the bismuth layer. A gamma autoradiograph of the aluminum backing of Target 59 after the bismuth layer had been removed is shown by Figure 4. The leading (north) edge of the target is still gamma active, but most of the gamma activity from the surface of the target, so prominent in Figure 3, is gone. On Target 62, bare aluminum was struck by the proton beam, producing a large yield of gamma activity in the aluminum impurities accompanied by a low alpha yield (47.5 per cent of the calculated yield) as shown in Figures 5 and 6.

"Superpure" (SP) aluminum was used as a backing material on four of the targets irradiated (Targets 46, 48, 55, and 56) Targets 46, 48 and 56 gave higher than average per cent alpha yields as well as low gamma yields, but Target 55 was just the reverse. However, Targets 27 and 49 whose backing tubes were made of 2S aluminum were similarly high in per cent alpha yield and low in gamma activity.

Purification of Cyclotron-Produced Polonium

Originally ORNL had planned to produce a target which would contain 100 millicuries of polonium. To conform with this plan, it was necessary to determine if the same purification scheme employed in the Operations Division would be effective at the fractional-curie level. Several products were prepared for electrolysis with fractions of a curie of polonium-210 as starting material. Yields of 70 per cent of the starting material could be obtained with a purity of 95 per cent, using as little as 0.66 curie of polonium-210 (the equivalent in weight to 85 millicuries of polonium-208). If recycling of the unpurified residues could be incorporated in the purifying scheme, then the fractional-curie targets could be processed individually. It was felt that processing the individual targets rather than the total curie as one product would minimize the chance of losing the material by a spill but might increase losses because of the repeated handling of material.

Process Method

The concentration hood is shown in Figure 7. Here the denitrated bismuth chloride solution of the target is drawn by vacuum from the reservoir, A, (of such a size as to

allow concentration of the material from several targets in one operation) through the line filter, B, over through the bed of bismuth powder, C, where the polonium is electrochemically deposited. The polonium-depleted bismuth chloride solution is collected in a suitable vessel at D. If necessary the electrochemical reduction operation could be repeated on the same or different powder.

Filter, C, containing the bismuth powder, was removed from the line and attached to reactor, E, in the purifying hood (Figure 8) in an inverted position. The bismuth was dissolved from the filter with a nitric-hydrochloric acid solution and drawn by vacuum into reactor, F.

This solution of the bismuth powder, containing in actual practice 35 - 300 millicuries of polonium-208, was denitrated and treated with stannous chloride.

After the reduced metal was redissolved, it was drawn by vacuum over to the reactor, F, (Figure 8) where it was treated with ammonium hydroxide to remove impurities which complexed with this reagent. After washing the insoluble polonium hydroxide was dissolved in 3 normal nitric acid, assayed for alpha activity, and transferred to the electrolysis hood (Figure 9). The polonium was plated from 1.5 normal nitric acid at the potential of the normal calomel electrode onto platinum microfoils held in the holder, G. The containers for the polonium plating solution H, calomel half-cells, J, and the stirring motor K, are shown in Figure 9. The polonium metal on the microfoils was weighed with a quartz-fiber balance, and the activity was determined either in a low-geometry counter or by calorimetry From weight and activity determinations, given in Table II, the per cent purity was calculated from the expression

$$\text{Per cent purity} = \frac{\text{Activity as determined by counting or calorimetry}}{\text{Activity as Po-208 (calculated from weight of deposit)}}$$

A flow sheet of analyses and process steps is given by Figure 10

The operations carried out in the concentration, purification and electrolysis hoods, as well as the mass-purity determination, are comparable to the corresponding higher-level operation described more fully in the Operation Division operation manuals.

Results of Purification of ORNL Polonium

The results of purification experiments are given in Table II. The numbers given in Column B refer to target numbers. Column D is the average mass-purity value derived by dividing the nitric acid solution of the purified polonium into three parts, plating a foil from each, assaying the plated polonium for purity, and taking the average of the three determinations.

The recovery and present disposition of the purified polonium-208 is given in Table III.

It is possible to purify as little as 33 millicuries of polonium-208 by the stannous chloride reduction method and to deliver as much as 78 per cent of this on foils (Run 6).

The purity values are consistent, yet they do not approach the purity values obtained with polonium-210. Self absorption in the samples counted in the low-geometry counter

was ruled out as a source of low purities when it was found that the amount of activity determined with the calorimeter and the counter agreed within 1.3 per cent for 40 millicurie foils or within 0.05 per cent for 26 foils containing 423 millicuries.

It is believed that a major cause of the low-purity values was the presence of the polonium-209 isotope, the specific activity of which is extremely low. Being chemically identical with polonium-208, polonium-209 had the effect of an inactive carrier and lowered the mass purity by an amount proportional to its weight. Two samples of polonium produced in the cyclotron were sent to Dr. I. Perlman at the University of California for isotopic purity determination. Preliminary results of the pulse-height analysis made on one sample indicate that the activity of the sample may be divided as follows:[5]

ISOTOPE	ACTIVITY, PER CENT	HALF LIFE
208	99.3	3 YEARS
209	0.2	200 YEARS
210	0.5	138 DAYS

If the ratio of the polonium-209 to polonium-208 half lives is multiplied by the activity due to the 209 isotope, it may be shown that about 13.5 per cent of the weight of polonium is attributable to the 209. This value agrees fairly well with the cross-section-energy (proton excitation) plot as well as the purity data given in Table II. If the half life of the 209 isotope is as great as 250 years, the weight of this isotope in our samples may be as much as 16.5 per cent. If this is true, the repurified sample (Run 5) which assays 83.2 per cent (without buoyancy correction) may be quite pure. It is reasonable to believe that the weighted purity average of 78.0 obtained for all the foils, is at least 90 per cent polonium.

The amount of material which had to be recycled is considerable. The sources of material requiring a rework are the stannous chloride reduction and the electrolysis residues. The fraction of the plating solution which was delivered on foils has increased from an average of 59 per cent for the first six runs to an average of 82 per cent for Runs 11 to 16. Preliminary study indicates that it will be possible to improve the recovery in the stannous chloride filtrate by allowing longer time for the reduced metal to coagulate before filtering and washing.

The recovery to date has been 71 per cent of the material received. A considerable fraction of the material has been lost in reworking. Some of the lost material should be recoverable when the purifying system is leached following this series of runs. As better technique is developed, undoubtedly there will be less material reworked, with less overall loss. The recovery of purified polonium at the fractional curie level is a rather tedious procedure at present.

Special Preparations

A spectrographic sample was prepared for analysis from the material from Run 5 (Table II). The polonium was volatilized from the platinum foils into a quartz tube two millimeters inside diameter and sealed in vacuum. This material (20.7 millicuries) was assayed as 83.2 per cent polonium-208 before volatilization.

Another sample of 76.6 per cent purity, 2.86 millicuries, was volatilized from a microfoil, sealed in an evacuated quartz tube, and sent to Dr. I. Perlman at the University of California for alpha-ray spectra analysis.

Half Life Study

Two samples of cyclotron-produced polonium are being counted periodically for a half-life study. The results after 202 days show that 86.6 per cent of the original material remains, whereas theoretically 87.7 per cent should remain if the material is polonium-208 with a half life of 2.93 years. These data are plotted in Figure 11.

Gamma Activity of ORNL Polonium

A half-thickness determination by gamma counting was made on a foil containing 44.3 millicuries of 84.4 per cent polonium-208. Two emitters were found, one with an energy of 0.87 and the other of 0.17 million electron volt (Figure 12). These results agree reasonably well with those of Perlman who found gammas of 0.86 and 0.55 million electron volt which he attributed to polonium-209, one per cent of which decays by gamma emission.[6] The gamma activity associated with 68 millicuries of polonium (808 counts per minute) was found to be 3.6 times as great as the activity calculated (222 counts per minute) if the entire polonium sample had been the 210 isotope.

Future Work

Purification experiments will continue on the ORNL targets as they are received and assayed. ORNL plans to produce approximately one curie per month for purification and study at Mound during the next six months.

After the one curie is sent to Los Alamos, the primary task will be to reprocess 100 millicuries to a maximum purity for a mass spectrographic determination to be carried out at Oak Ridge. The alpha-activity distribution in the targets will also be studied more thoroughly using a combination of the autoradiographs and a target-sectioning technique.

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1. Nelson, G. D. and Fox, G. L., Rpt. Gen. Res., MLM-597, p. 31, August 7, 1951.
2. Nelson, G. D. and Fox, G. L., Rpt. Gen. Res., MLM-559, p. 5, May 21, 1951.
3. Monthly Status Letter from Livingston, R. S. to Haring, M. M., (51-9-11)
4. Stetson, N., Private Communication, October 1, 1951.
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6. DeBennedetti, et al., "The Measurement of Polonium by Gamma Rays", Final Report No. 23, January 1, 1946.

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TABLE I

ASSAY OF ORNL TARGETS 35 - 69

TARGET	SURFACE AREA (in.)	ESTIMATED ALPHA YIELD (mC.)	BISMUTH (g.)	ALPHA ASSAY (mC.)	GAMMA ASSAY (mC.)	PER CENT OF ESTIMATED ALPHA YIELD	MC. γ PER 100 MC. α	NOTES
35	2 x 6	70.7	10.4	42.7	2.54	60.2	6.00	
36	2 x 6	16.4	13.5	12.1	0.47	73.4	3.89	
24	2 x 6	0.92	10.1	0.90	0.02	97.8	1.67	
38	2 x 6	0.95	20.2	0.72	0.01	76.6	0.97	
27	2 x 6	1.01	20.9	0.80	0.004	79.2	0.50	
28	2 x 6	0.63	15.4	0.40	0.005	63.5	1.25	
44	6 x 7	10.5	40.2	6.76	0.072	64.4	1.07	
46	2 x 6	1.06	12.9	1.12	0.002	105.9	0.18	S.P. ALUMINUM
47	5 x 6	12.1	40.6	6.57	0.109	59.7	1.66	
48	2 x 6	1.03	5.5	0.87	0.003	84.3	0.34	S.P. ALUMINUM
45	6 x 7	14.0	60.0	8.2	0.085	58.7	1.04	
49	5 x 6	3.58	35.2	2.27	0.022	63.3	0.97	
50	5 x 6	23.0	38.0	17.3	0.268	75.2	1.59	ALUMINUM VISIBLE THROUGH BISMUTH.
53	5 x 6	70.1	44.0	42.8	0.577	61.0	1.35	PINS ADDED TO PREVENT BULGING.
54	6 x 7	20.4	72.3	5.02	0.224	24.6	4.46	
55	2 x 6	6.24	15.0	3.00	0.247	48.1	8.23	S.P. ALUMINUM
56	2 x 6	4.65	15.0	3.81	0.024	82.0	0.63	S.P. ALUMINUM
57	6 x 7	17.5	42.9	9.45	0.278	54.1	2.94	
58	5 x 6	85.1	76.2	37.4	1.74	44.1	4.65	
59	6 x 6	221.4	44.0	153.0	2.94	69.3	1.92	
60	6 x 6	2.85	57.7	1.91	0.105	67.0	5.50	
61	6 x 6	23.3	21.0	10.9	0.551	47.0	5.06	
62	6 x 6	219.2	34.0	104.0	5.29	47.5	5.05	ALUMINUM VISIBLE THROUGH BISMUTH.
63	6 x 6	47.6	25.0	19.8	2.51	41.6	12.6	
64	6 x 6	280.0	44.0	145.0	5.89	51.8	4.06	
65	6 x 6	288.3	32.4	146.5	2.72	50.6	1.86	
66	6 x 6	99.0	39.4	28.7	4.53	28.0	15.8	
69	6 x 10	488.9	54.5	181.0	9.17	37.0	5.07	

RECEIVED PREVIOUSLY 56.9 MC.

TOTAL FROM 35 - 69 = 992.4

TOTAL RECEIVED THROUGH SEPTEMBER 30 = 1049.3 MC.

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TABLE II

PURIFICATION OF ORNL POLONIUM

A	B		C	D	E	F		G		H	
PRODUCT	ORIGINAL MATERIAL (Source)	(mC.)	ON MICRO FOILS (mC.)	AVERAGE PURITY (%)	ON FOILS (%)	RECYCLE (mC.)	RECYCLE (% of orig.)	RECOVERY PER CENT** (Run)	RECOVERY PER CENT** (Accum.)	ACCUMULATIVE ACTIVITY RECEIVED (mC.)	ACCUMULATIVE ACTIVITY ON FOILS (mC.)
1	35, 36	57.1	18.66	76.3	32.6	34.0	59.5	92.1	92.1	57.1	
2	RECYCLE 1 8 - 34	88.4	28.15	46.7	31.8	17.3	19.7	51.4	57.4	111.7	
3	RECYCLE 1 AND 2	51.3	20.92	67.2	40.7	17.4	33.9	74.7	76.2		
4	24 - 48 PLUS RECYCLE 3	35.54	23.15	67.7	65.0	0.9	2.5	67.7	70.7	129.8	
5	REWORK 1 - 4	90.88	20.70	83.2	22.8	32.6	35.9	58.6	41.1		20.7
6	RECYCLE 5	32.60	25.56	82.8	78.4	17.5	53.4	132.1	49.1		46.26
7	RECYCLE 4,6 45 - 50	45.30	26.36	80.0	56.0	23.4	51.7	109.9	61.3	156.73	72.62
8	RECYCLE 7 53 - 58	125.2	39.50	72.2	31.6	40.7	32.5	64.1	59.1	258.5	112.12
9*	RECYCLE 8	40.7	3.07	55.0	7.6	-	0	7.6	44.6		
10	59,60,61	147.5	37.42	76.5	25.4	120.2	81.5	106.9	64.3	424.3	149.54
11	SCAVENGE	120.2	64.39	78.2	53.5	36.0	30.0	83.5	64.3		213.93
12A	RECYCLE 11 62,63,64	304.0	140.18	79.2	56.5	128.0	42.1	75.0	74.5	693.1	
12B			31.22	75.9							385.33
13	RECYCLE 12	128.0	40.55	78.8	31.8	49.7	38.8	70.5	69.1		425.88
14	RECYCLE 13 PRODUCT 9	49.7	17.22	74.9	34.8	67.0	169.5	169.5	73.6		443.10
15A	RECYCLE 14 65,66,69	423.0	103.15	76.6	49.5	143.0	33.8	83.3	75.9	1049.3	652.39
15B			106.14	77.7							
16	RECYCLE 15	143.0	37.64	79.5	26.3	76.1	53.2	79.5	73.1		690.03
17	RECYCLE 16	76.1	8.6	72.5	11.3	49.4	64.9	76.2	71.4		698.63
18	RECYCLE 17	49.4									

AVERAGE PURITY, 78.0 PER CENT

* NOT INCLUDED IN TOTALS
** INCLUDES DECAY LOSSES

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TABLE III

RECOVERY OF PURIFIED ORNL POLONIUM

	MILLICURIES	PER CENT
TOTAL RECEIVED THROUGH 9/30/51	1049.3	100
DELIVERED ON FOILS (FINAL PRODUCT)	678.0	64.7
TO TIMMA FOR SPECTROGRAPHIC ANALYSIS	20.7	2.0
IN PROCESSING	49.4	4.7
SENT TO I. PERLMAN	2.9	0.3
SET ASIDE (CONTAINS PLATINUM)	4.6	0.4
APPARENT PROCESSING LOSS	292.7	27.9

ALL ACTIVITIES AS OF OCTOBER 1, 1951

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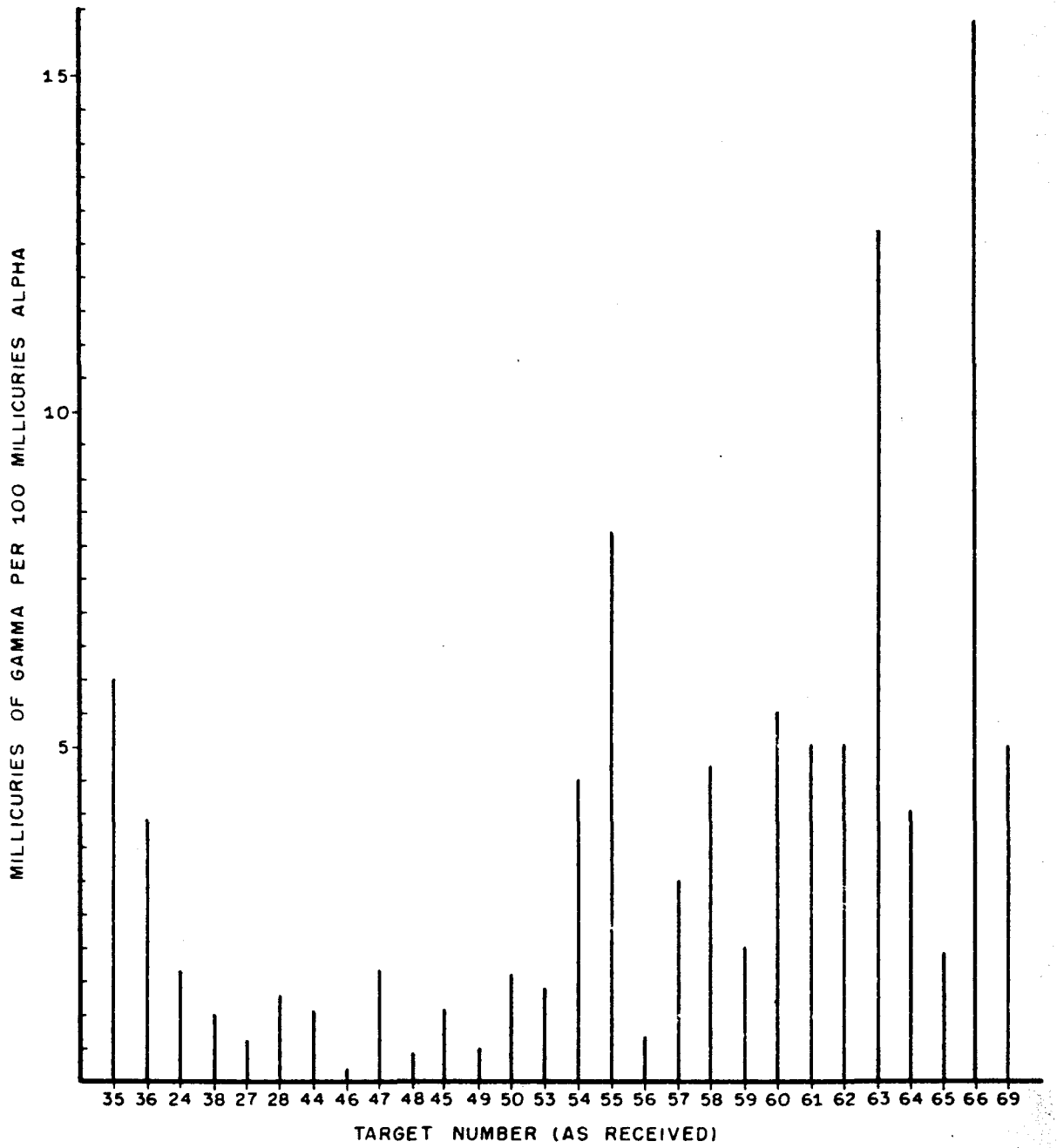

GAMMA ACTIVITY IN ORNL TARGETS 35-69

5094

FIGURE 2

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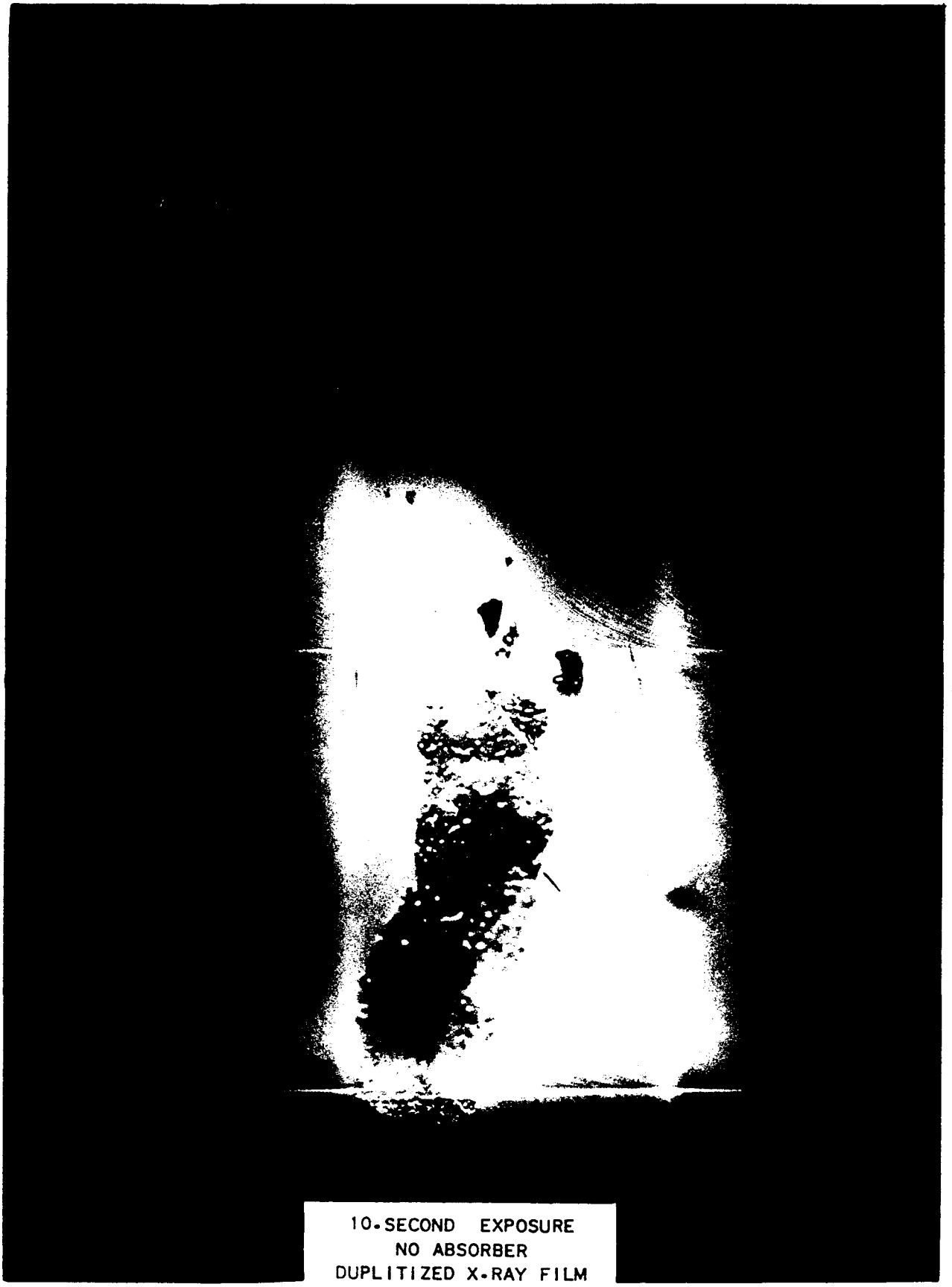

10-SECOND EXPOSURE
NO ABSORBER
DUPLITIZED X-RAY FILM

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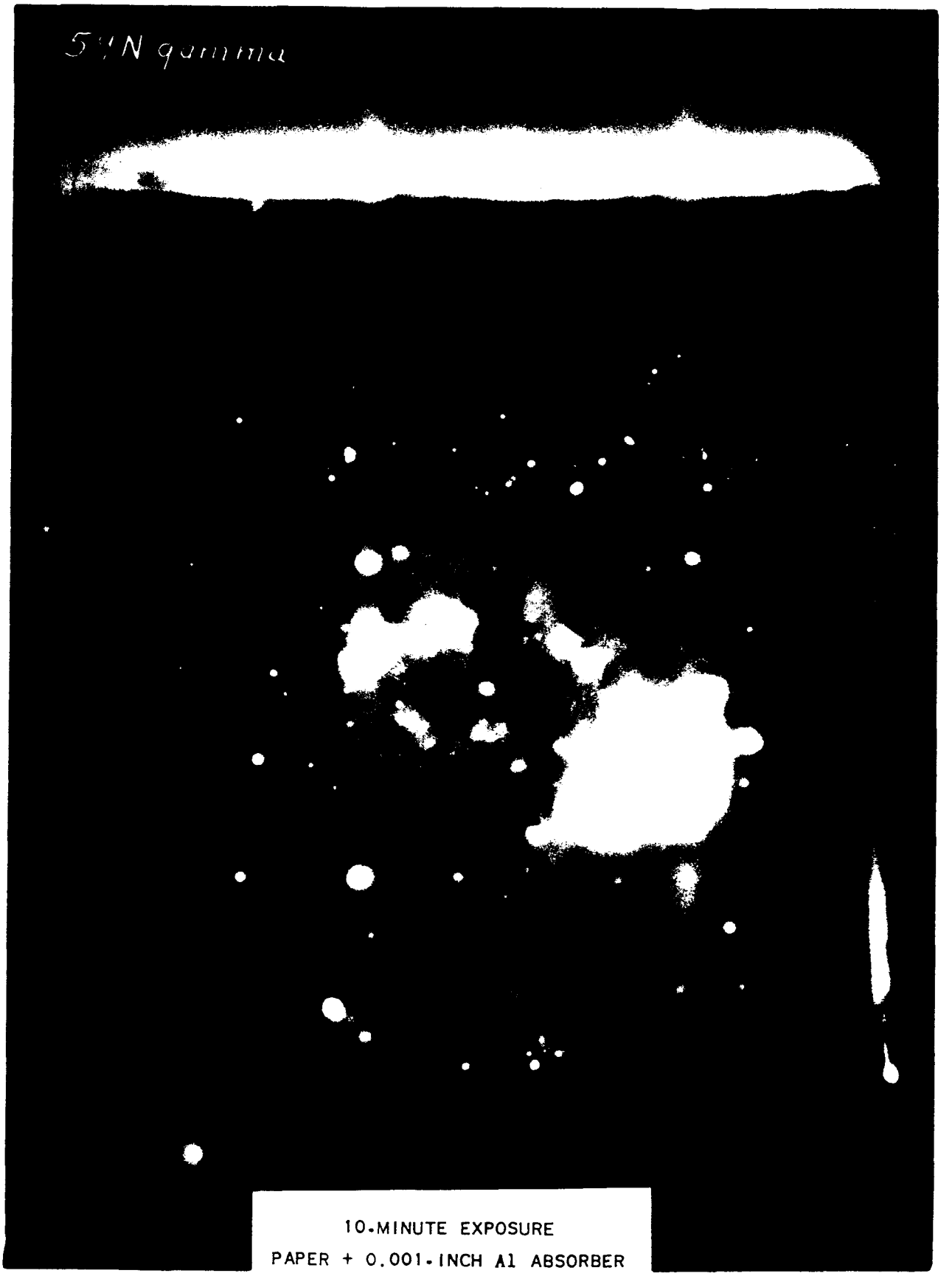
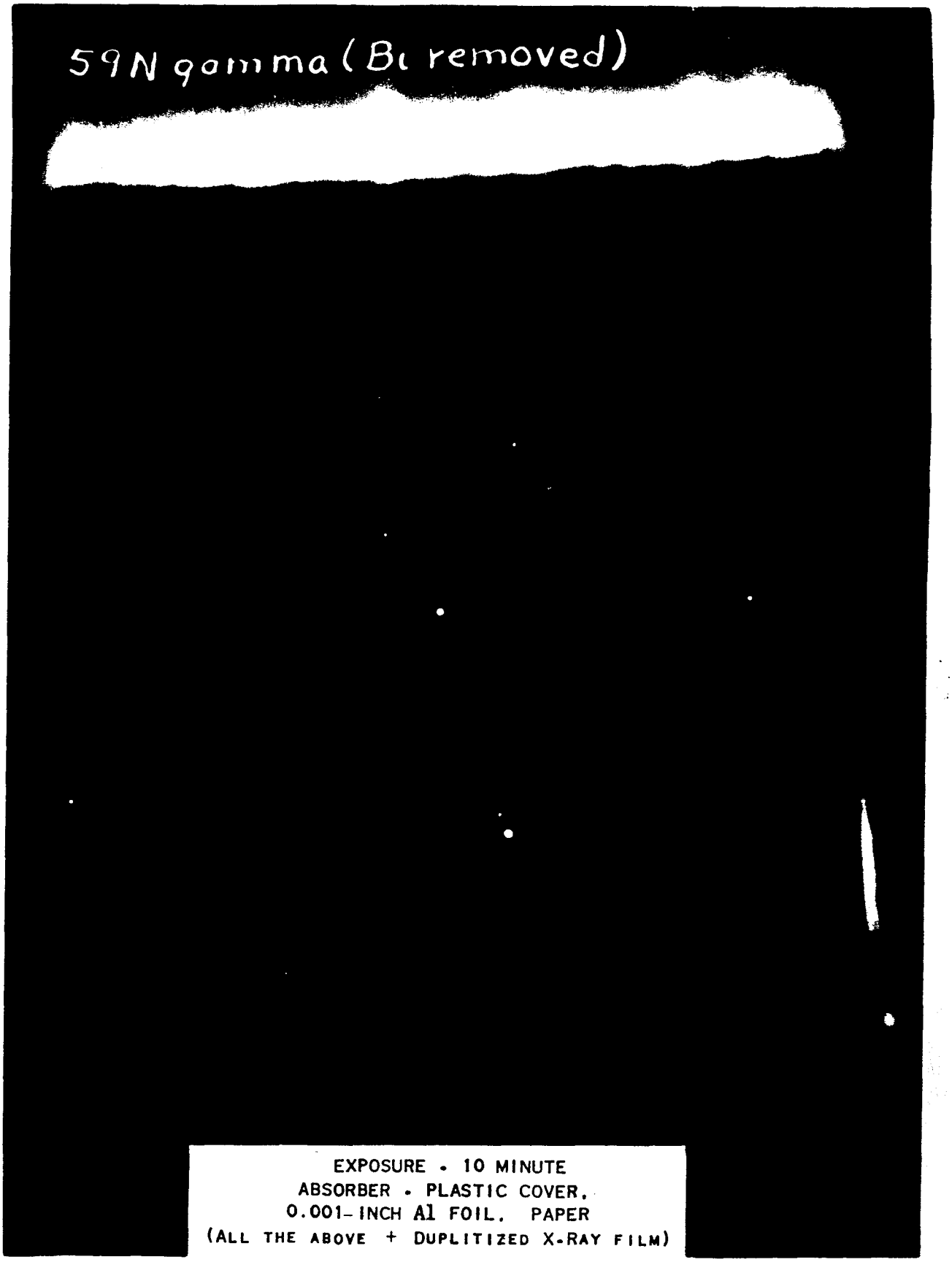
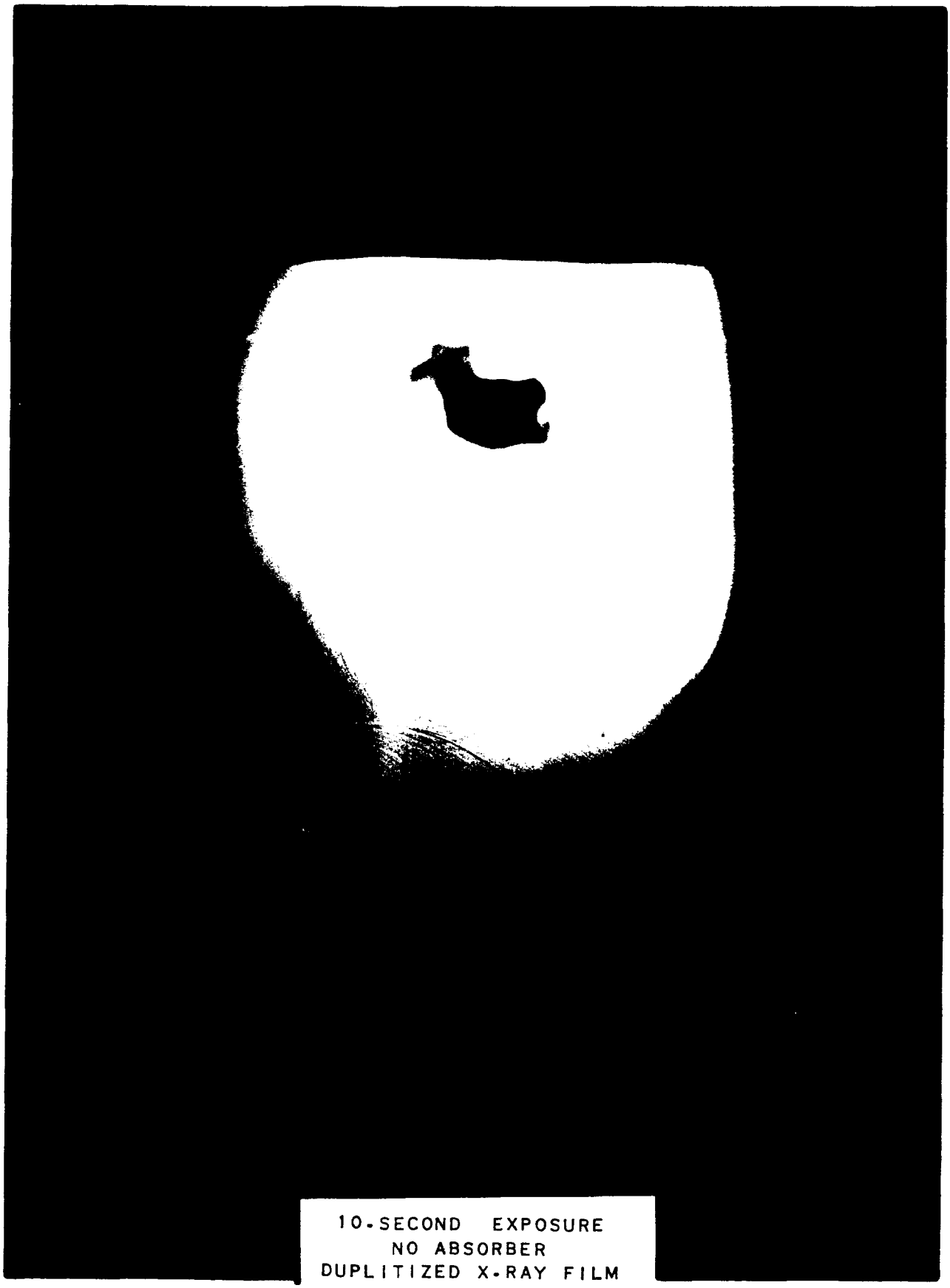


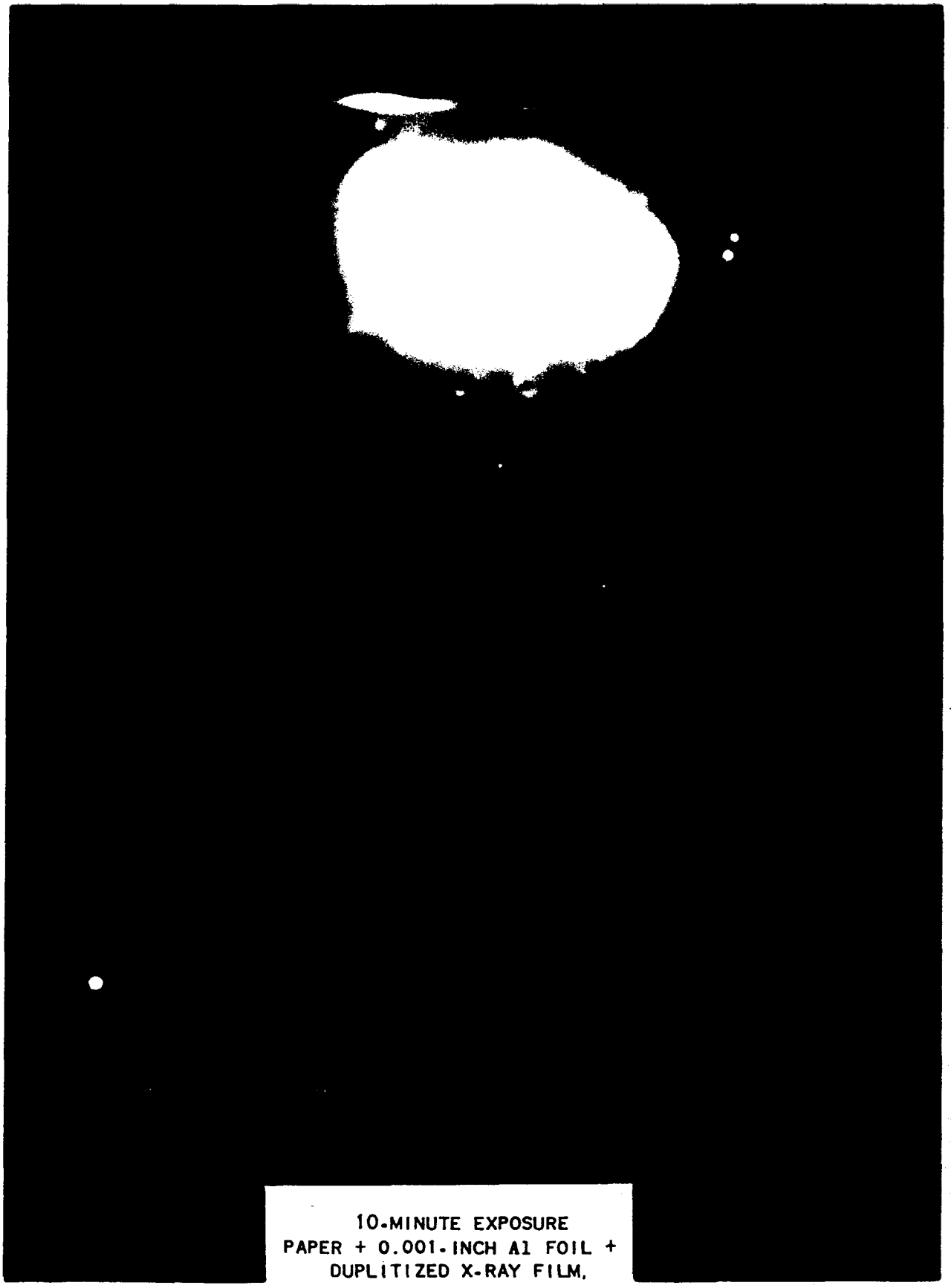
10-MINUTE EXPOSURE
PAPER + 0.001-INCH Al ABSORBER



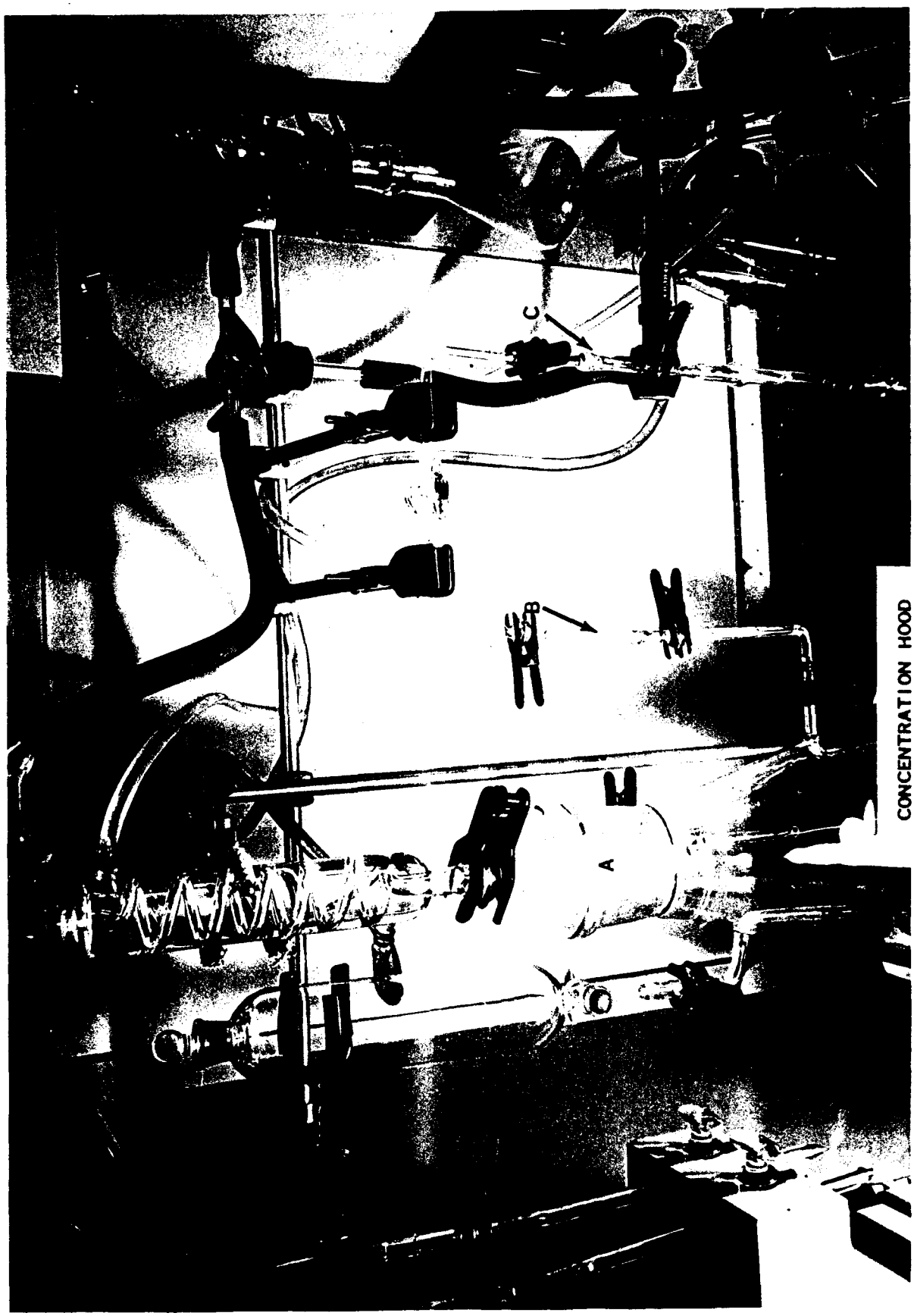
EXPOSURE - 10 MINUTE
ABSORBER - PLASTIC COVER,
0.001-INCH Al FOIL, PAPER
(ALL THE ABOVE + DUPLITIZED X-RAY FILM)



10-SECOND EXPOSURE
NO ABSORBER
DUPLITIZED X-RAY FILM

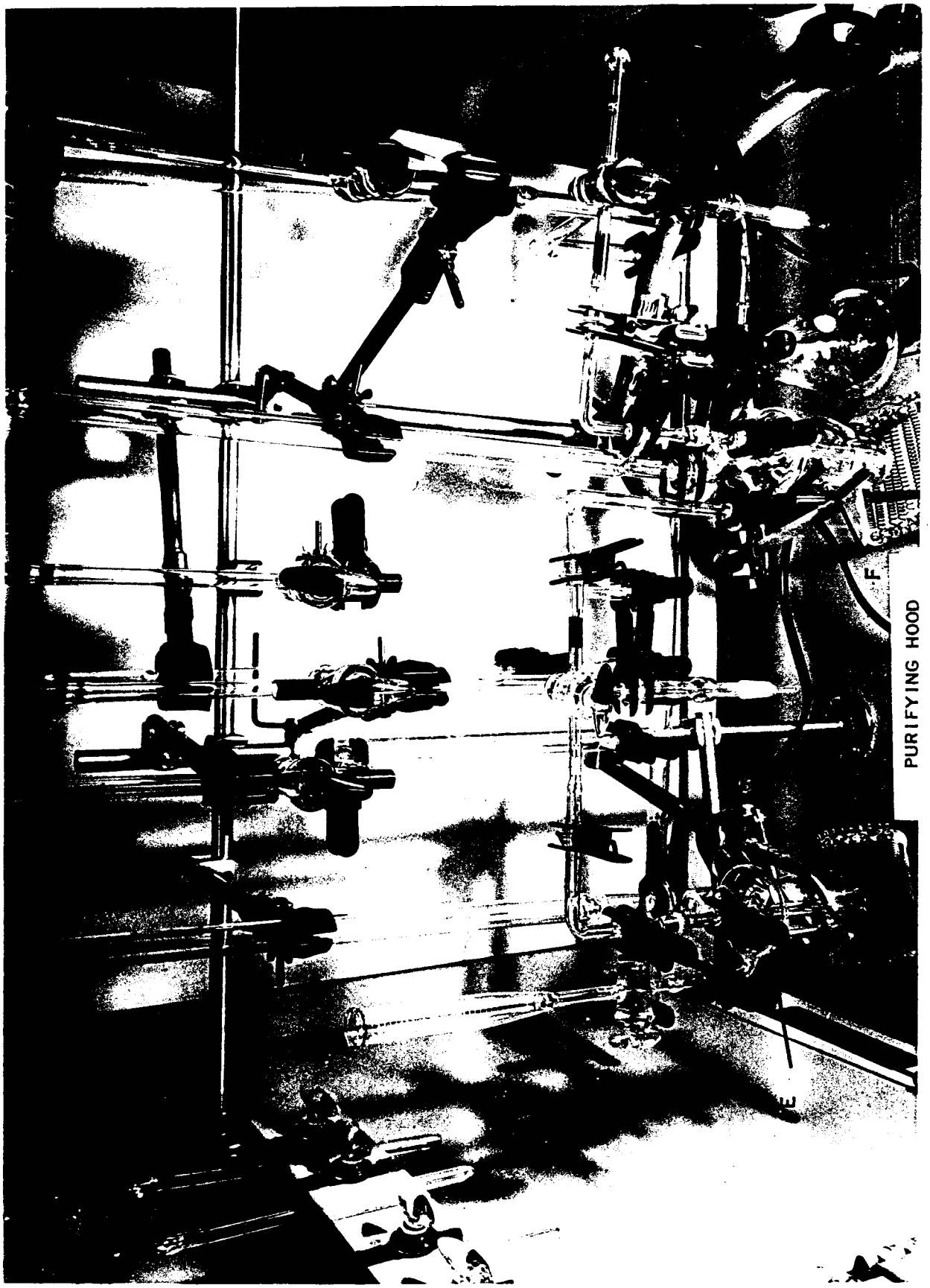


10-MINUTE EXPOSURE
PAPER + 0.001-INCH Al FOIL +
DUPLITIZED X-RAY FILM.



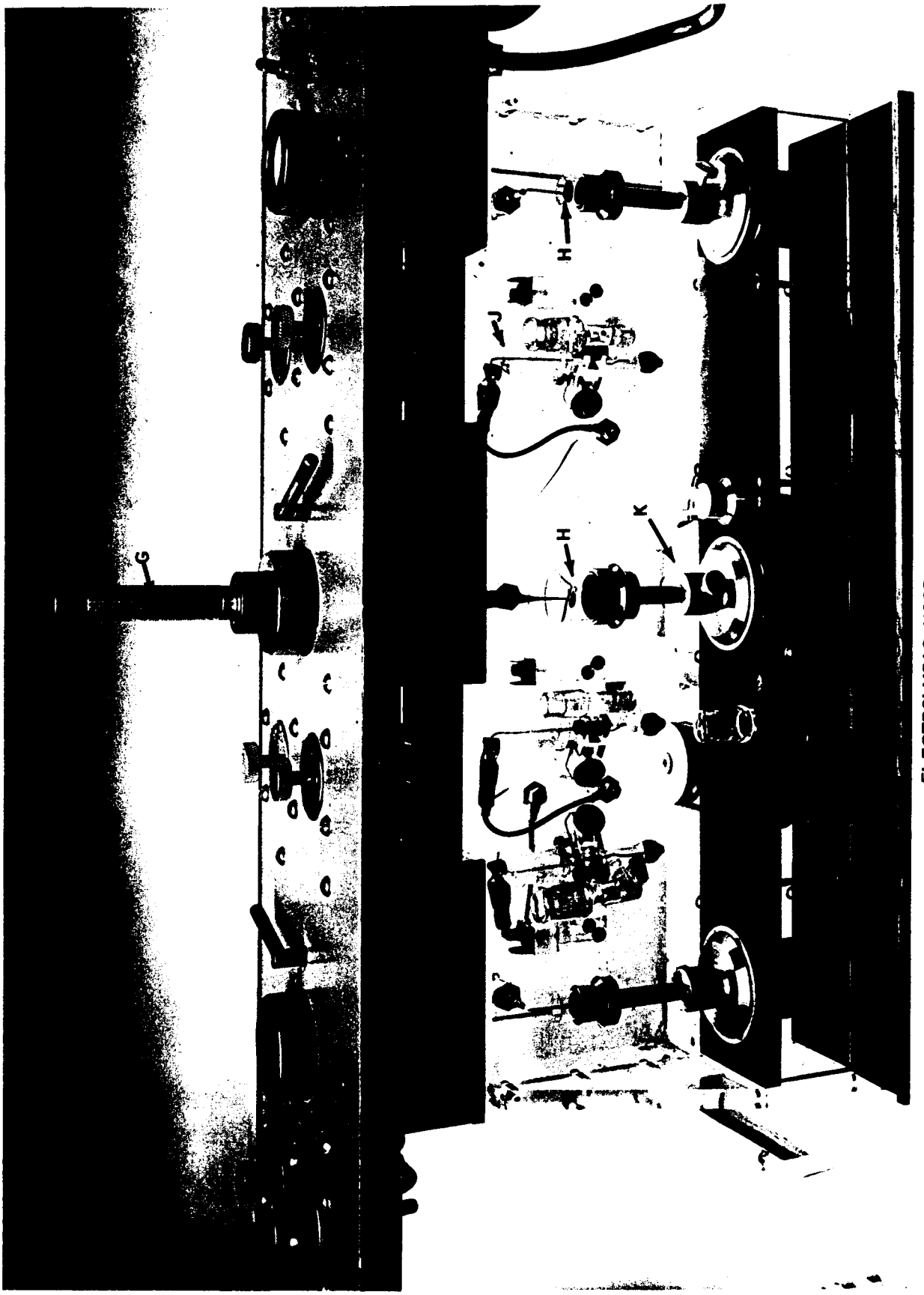
CONCENTRATION HOOD

5
1
0
3
8
9



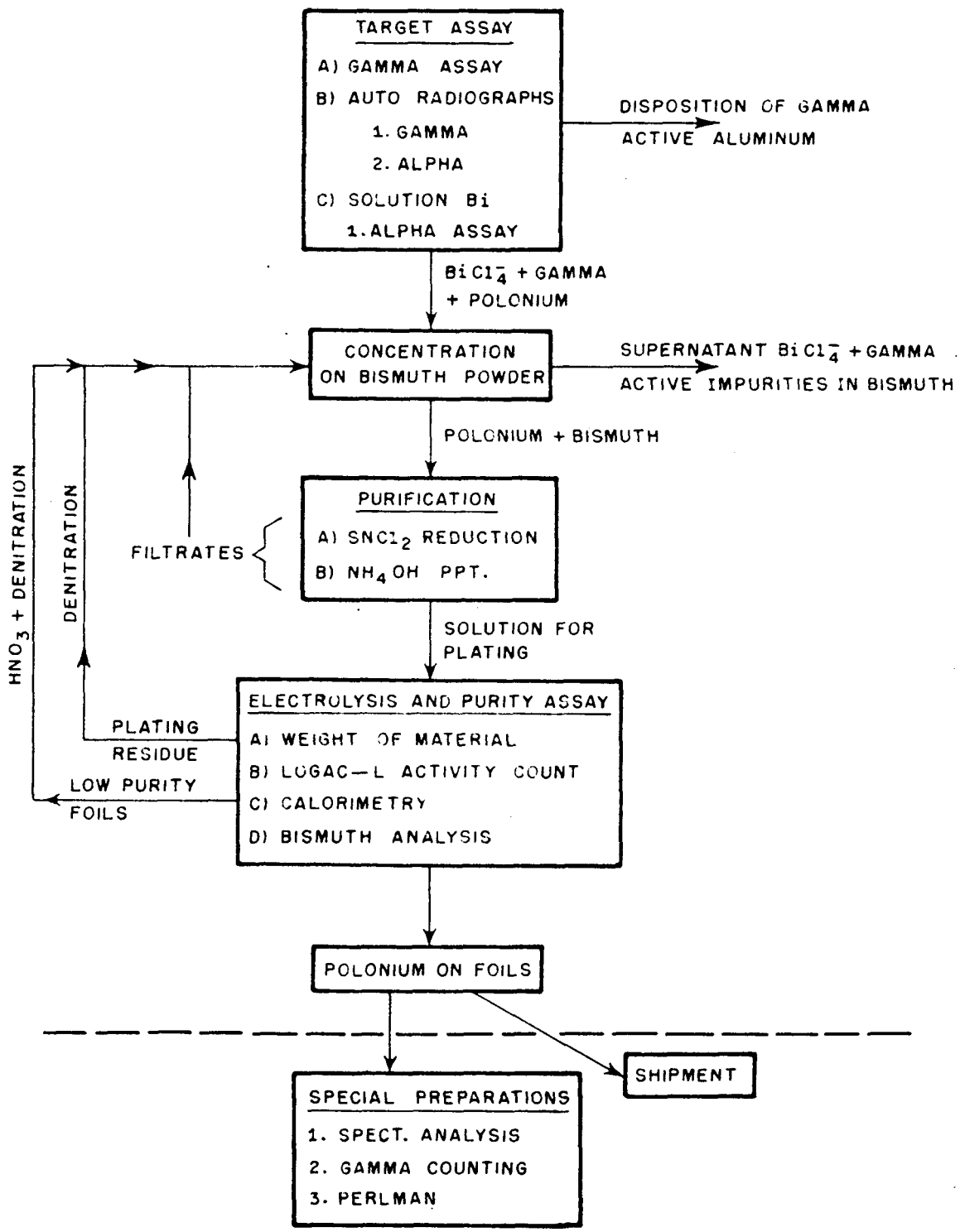
PURIFYING HOOD

5
1
0
3
9
0



ELECTROLYSIS HOOD

5
1
0
3
9
1

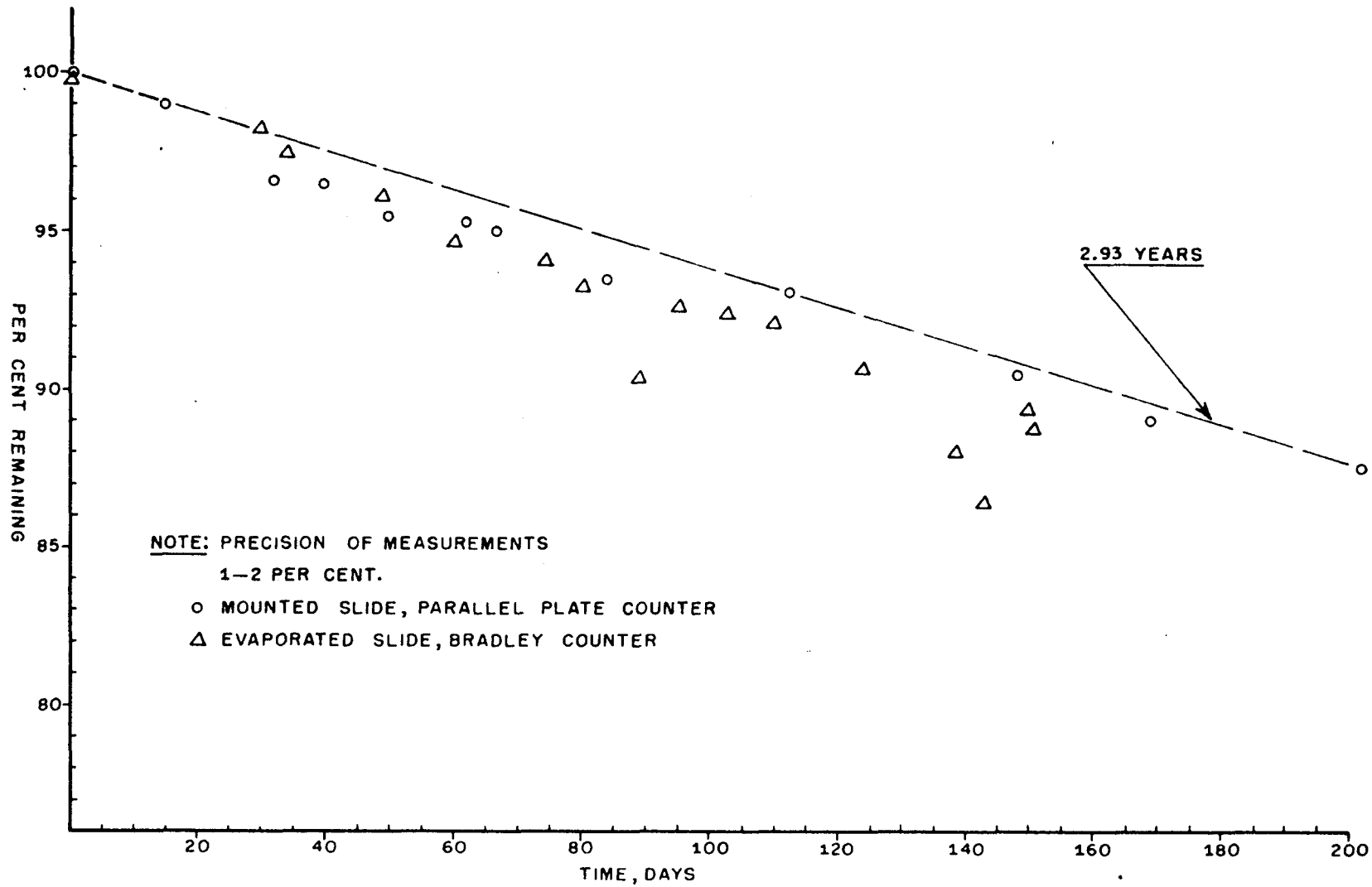


FLOW OF ORNL POLONIUM DURING ASSAY AND PURIFICATION.

5092

617-6A-11

5093



HALF LIFE STUDY OF ORNL POLONIUM.

FIGURE 11

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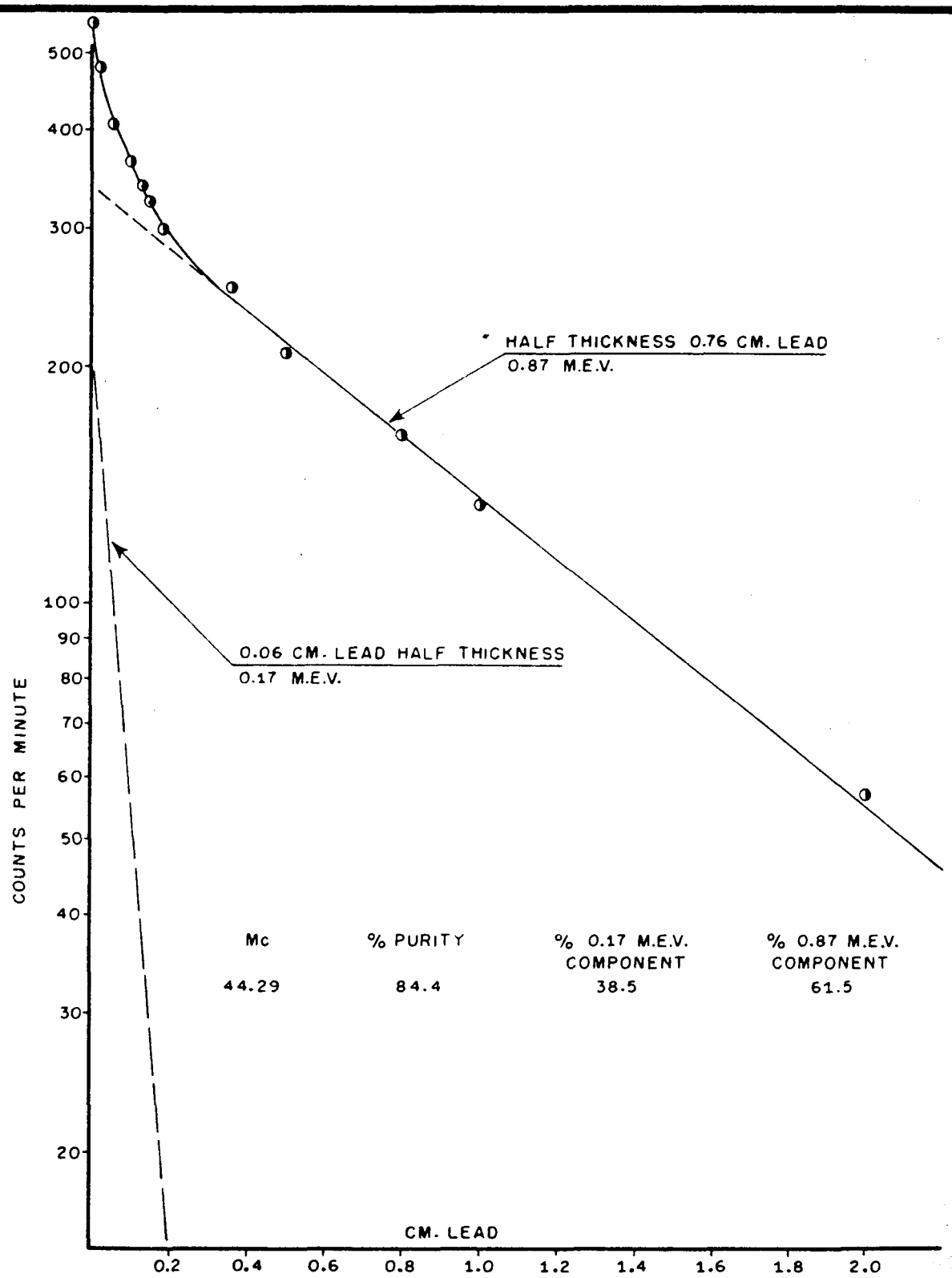

HALF THICKNESS OF GAMMA EMITTERS FROM PURIFIED ORNL POLONIUM.

5095

Problem Title -- **Polonium Migration in a High Vacuum under Isothermal Conditions**

Report By -- **W. C. McCluggage**

Work Done By -- **W. C. McCluggage**

INTRODUCTION

The probability of polonium contamination in a cyclotron or a linear accelerator has indicated a need for data on polonium migration in a vacuum from a spot source in the absence of a temperature gradient. To obtain these data, a series of experiments was designed in which a polonium source was exposed in a vacuum chamber. The chamber was so constructed that a removable liner could be checked for contamination.

DETAILED REPORT

Construction of Equipment

The all-aluminum apparatus, equipped with a removable aluminum liner and a base plate to which the polonium sample was attached, is shown in Figures 1 and 2. Aluminum was chosen because it is easily decontaminated with concentrated nitric acid. The cylinder was machined from a piece of round stock. To improve the vacuum seal, the base plate was equipped with a "V" recess near the edge into which a ridge on the cylinder flange fitted. The top of the cylinder contained a threaded shoulder fitted with a half-inch pipe which led to the high-vacuum system. The cylinder liner, a baffle plate at the opening to the vacuum system, and the gasket covering the base plate were made of 0.012-inch sheet aluminum.

Experimental

The polonium sources which had a ¼-inch spot of polonium in the middle of a metal disk, were prepared in two ways. For Run No. 1 the source was prepared by electroplating about 160 millicuries from a 1.5-normal nitric acid solution onto a 2-inch nickel disk. A potential of -0.4 volt with respect to a saturated calomel cell was used. All other sources were obtained by evaporating polonium onto 2-inch aluminum disks which, with the exception of the ¼-inch spot in the center, were covered by a protective coating. These sources all contained about 160 millicuries.

The polonium-bearing disks were fixed to the base plate with four small screws. The plate was fastened to the cylinder with four ¼-inch bolts. To assure a better seal, silicone vacuum grease was used on the metal gasket.

Five runs were made; four in vacuo and one at atmospheric pressure (Table I). Immediately following each run, the apparatus was disassembled. The top liner disk, the baffle plate, and the side-wall liner were autoradiographed for visual observation of the polonium distribution. A typical picture is shown by Figure 3. The contamination appears to be the result of small particles of polonium blown from their source by air currents instead of by vaporization of the metal. After the autoradiographs were obtained, the side-wall liner was cut into six equal slides and numbered as shown in Figure 2. These slides,

the top liner disk, and the baffle plates were placed in rubber hydrochloride envelopes and the alpha activity determined by ion current measurements. Corrections for absorption of the rubber hydrochloride film were made. Results for this work are given in Tables II to VI.

CONCLUSIONS

At room temperature and atmospheric pressure (Run No. 5), no appreciable contamination of the cylinder liner was detected after 18 hours exposure. At ice-water temperature and a pressure of 4×10^{-6} millimeter of mercury, the average contamination on the liner was equivalent to 3.8×10^{5} disintegrations per minute per square centimeter. This value increased to 6.4×10^{5} at room temperature and to 1.7×10^{6} at 87°C. In Run No. 4 conducted at room temperature and for only two hours (time required to obtain a pressure of 1.0×10^{-5} millimeter of mercury) the average contamination was 3.4×10^{5} disintegrations per minute per square centimeter.

The contamination levels indicate that the evacuation of the apparatus is the principal cause of the spread of activity. Time and temperature contribute to the contamination build up (Tables II to VI).

From the autoradiograph (Figure 3) it appears that currents of air or the degassing of the polonium source distribute minute particles in the vacuum chamber. These particles are the major cause of the contamination.

TABLE I

CONDITIONS FOR STUDY OF POLONIUM MIGRATION

RUN NO.	TEMPERATURE (°C.)	PRESSURE (mm. Hg)	DURATION (hr.)
1	4	4×10^{-6}	18
2	25	4×10^{-6}	18
3	87	1×10^{-5}	18
4	25	1×10^{-5}	2
5	25	ATMOSPHERIC	18

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TABLE II

MIGRATION OF POLONIUM

RUN NO. 1

TEMPERATURE	4°C ± 2°
PRESSURE	4.0×10^{-6} MM. HG
ELAPSED TIME	18 HOURS
POLONIUM CONTENT OF SAMPLE	160 MILLICURIES
SAMPLE PREPARATION	ELECTRODEPOSITION ON NICKEL*

LOCATION	AREA COUNTED (sq. cm.)	α COUNT (DIS./MIN./CM.²) EXPOSED SIDE	BACK SIDE
BAFFLE PLATE	4.9	1.15×10^6	3.76×10^5
TOP LINER DISK	14.7	3.50×10^5	6.62×10^4
SIDE-WALL LINER			
SEGMENTS 1	12.5	3.58×10^5	3.34×10^4
2	12.5	4.65×10^5	2.33×10^4
3	12.5	4.65×10^5	2.46×10^4
4	12.5	3.26×10^5	2.46×10^4
5	12.5	3.33×10^5	2.08×10^4
6	12.5	3.73×10^5	1.72×10^4

* SAMPLE 1 ONLY WAS PREPARED ELECTROLYTICALLY.

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TABLE III

MIGRATION OF POLONIUM

RUN NO. 2

TEMPERATURE	25°C ± 2°
PRESSURE	4.0×10^{-6} MM. HG
ELAPSED TIME	18 HOURS
POLONIUM CONTENT OF SAMPLE	165 MILLICURIES
SAMPLE PREPARATION	METAL WAS EVAPORATED ONTO ALUMINUM DISK

LOCATION		AREA COUNTED (sq. cm.)	α COUNT (DIS./MIN./CM.²) EXPOSED SIDE	BACK SIDE
BAFFLE PLATE		4.9	3.43×10^6	4.77×10^6
TOP LINER DISK		14.7	1.06×10^6	5.35×10^5
SIDE-WALL LINER				
SEGMENTS	1	12.5	6.10×10^5	1.65×10^5
	2	12.5	5.92×10^5	1.65×10^5
	3	12.5	6.48×10^5	1.58×10^5
	4	12.5	6.10×10^5	6.30×10^5*
	5	12.5	4.85×10^5	1.81×10^5
	6	12.5	4.75×10^5	1.65×10^5

* THIS SEGMENT OF THE SIDE-WALL LINER WAS NOT HELD FIRMLY AGAINST THE CYLINDER WALL.

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TABLE IV

MIGRATION OF POLONIUM

RUN NO. 3

TEMPERATURE	87°C ± 2°
PRESSURE	1.0×10^{-5} MM. HG
ELAPSED TIME	18 HOURS
POLONIUM CONTENT OF SAMPLE	175 MILLICURIES
SAMPLE PREPARATION	METAL EVAPORATED ONTO ALUMINUM DISK

LOCATION		AREA COUNTED (sq. cm.)	α COUNTS (DIS./MIN./CM.²) EXPOSED SIDE	BACK SIDE
BAFFLE PLATE		4.9	1.02×10^6	1.37×10^6
TOP LINER DISK		14.7	1.17×10^6	4.76×10^5
SIDE-WALL LINER				
SEGMENTS	1	12.5	1.39×10^6	3.86×10^5
	2	12.5	1.58×10^6	1.90×10^5
	3	12.5	1.86×10^6	2.69×10^5
	4	12.5	1.86×10^6	1.77×10^5
	5	12.5	1.91×10^6	1.20×10^5
	6	12.5	2.00×10^6	1.60×10^5

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TABLE V

MIGRATION OF POLONIUM

RUN NO. 4

TEMPERATURE	25°C ± 2°
PRESSURE	1.0×10^{-5}
ELAPSED TIME	2 HOURS
POLONIUM CONTENT OF SAMPLE	160 MILLICURIES
SAMPLE PREPARATION	METAL EVAPORATED ONTO ALUMINUM DISK

LOCATION		AREA COUNTED (sq. cm.)	α COUNT (DIS./MIN./CM.²) EXPOSED SIDE	BACK SIDE
BAFFLE PLATE		4.9	4.40×10^5	2.38×10^5
TOP LINER DISK		14.7	3.53×10^5	2.44×10^4
SIDE-WALL LINER				
SEGMENTS	1	12.5	4.66×10^5	7.10×10^3
	2	12.5	4.15×10^5	6.68×10^3
	3	12.5	3.70×10^5	5.85×10^3
	4	12.5	2.94×10^5	1.04×10^4
	5	12.5	2.28×10^5	6.25×10^3
	6	12.5	2.36×10^5	5.42×10^4

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TABLE VI

MIGRATION OF POLONIUM

RUN NO. 5

TEMPERATURE	25°C ± 2°
PRESSURE	ATMOSPHERIC
ELAPSED TIME	18 HOURS
POLONIUM CONTENT OF SAMPLE	160 MILLICURIES
SAMPLE PREPARATION	METAL EVAPORATED ONTO ALUMINUM DISK

LOCATION		AREA COUNTED (sq. cm.)	α COUNT (DIS./MIN./CM.²) EXPOSED SIDE	BACK SIDE
BAFFLE PLATE*				**
TOP LINER DISK		19.6	84	
SIDE-WALL LINER				
SEGMENTS	1	12.5	174	
	2	12.5	33	
	3	12.5	80	
	4	12.5	8	
	5	12.5	10	
	6	12.5	57	

* NO EVACUATION TOOK PLACE SO THE BAFFLE PLATE WAS OMITTED.

** BECAUSE OF THE LOW ORDER OF CONTAMINATION THE BACKS OF THE SLIDES WERE NOT COUNTED.

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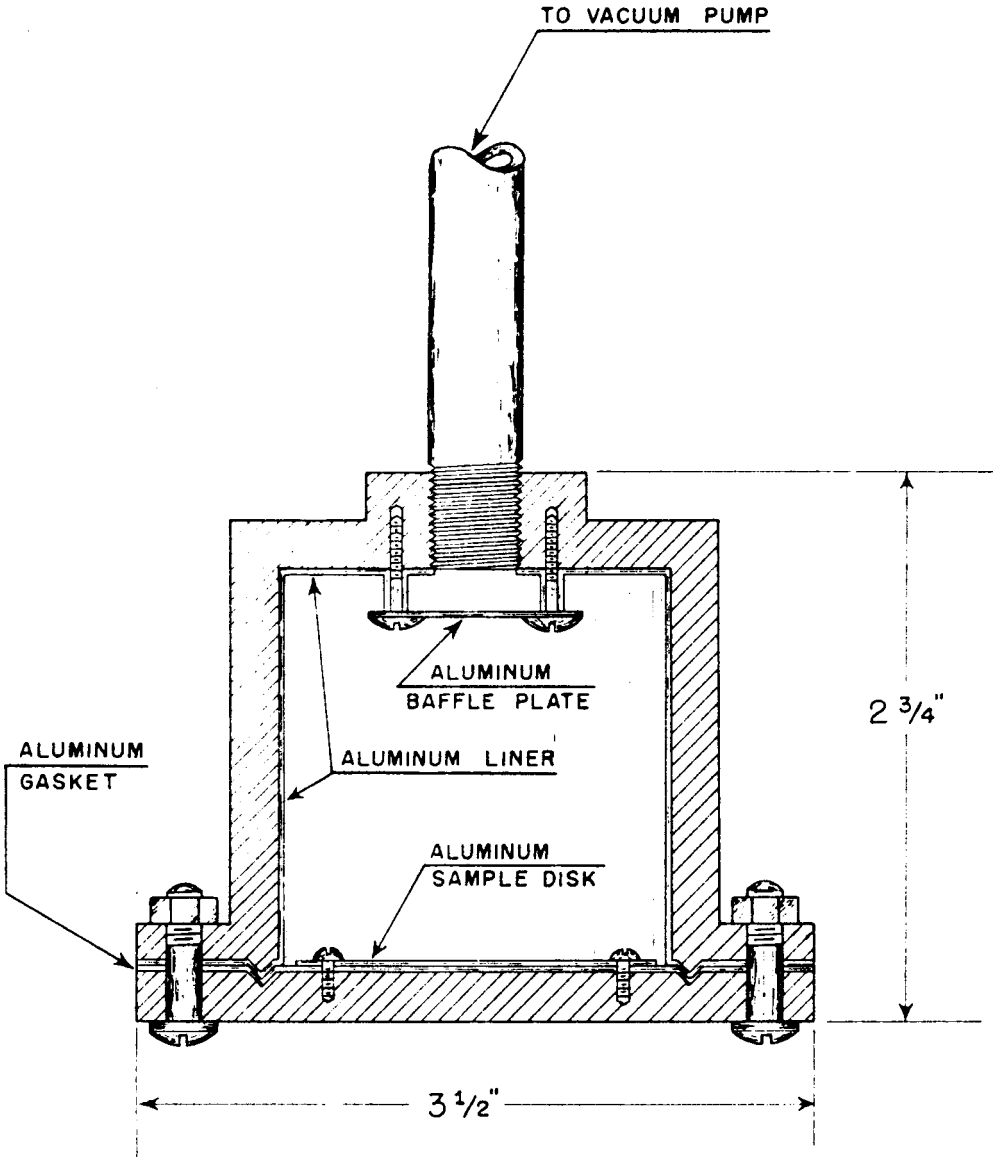
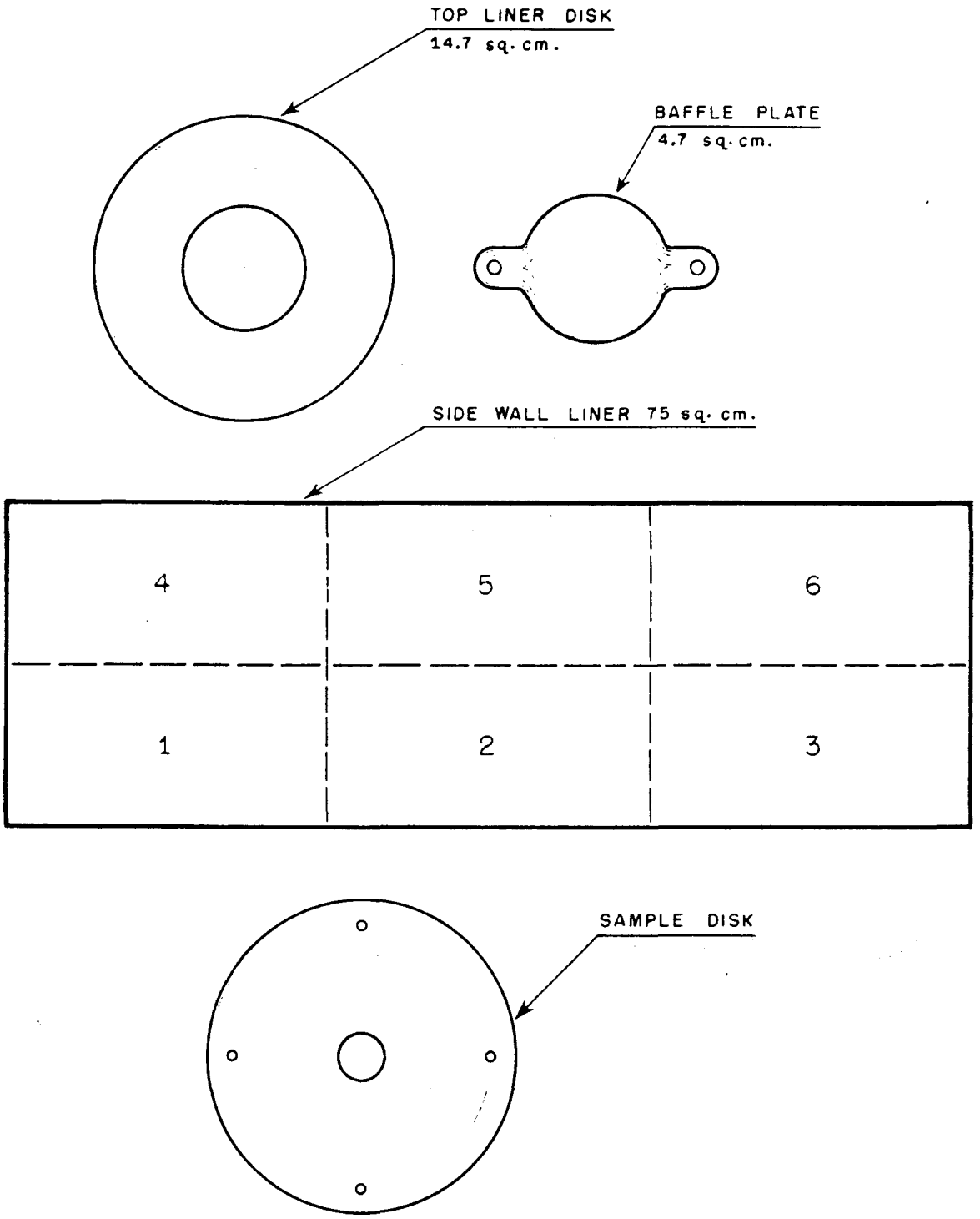


NOTE: ALUMINUM BAFFLE PLATE, LINER AND SAMPLE DISK ARE MADE OF 0.012" STOCK.

5091

CYLINDER FOR STUDY OF POLONIUM SCATTERING IN VACUUM.

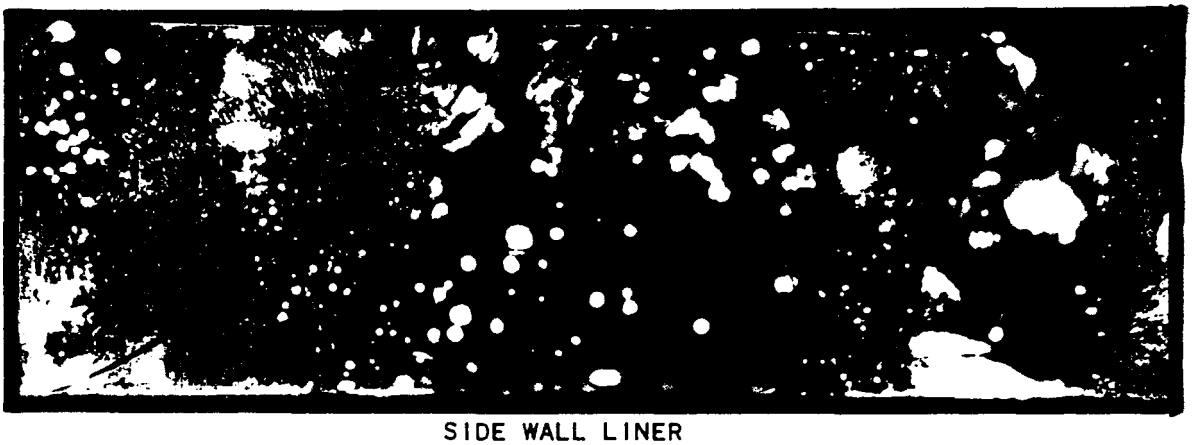


ALUMINUM LINER,
BAFFLE PLATE AND SAMPLE DISK.

5090

TOP LINER DISK

BAFFLE PLATE



SIDE WALL LINER

NOTE: BLACK LINES CAUSED BY WRINKLES IN THE RUBBER HYDROCHLORIDE ENVELOPE.

AUTORADIOGRAPH OF CYLINDER LINER AND BAFFLE PLATE

Problem Title -- **Loss of Polonium from an Uncovered Bismuth-Polonium Surface**

Report By -- **J. B. Otto**

Work Done By -- **J. B. Otto**

INTRODUCTION

The determination of the loss of polonium from bismuth-polonium surfaces is of importance in connection with the design of targets for cyclotrons and linear accelerators producing polonium-208 by bombardment of bismuth with protons. The loss of appreciable quantities of polonium from such targets might seriously contaminate associated apparatus. The present experiments were carried out to determine the loss of polonium from an uncovered bismuth-polonium surface. Other studies with the same apparatus are proposed to determine the loss of polonium from bismuth-polonium surfaces covered with thin layers of various protective coatings.

DETAILED REPORT

The apparatus and preparation of sample used in these experiments has been described previously.[1] Since the sample used in this work was cast from molten bismuth-polonium, the polonium was not homogeneously distributed in the surface but was slightly more concentrated at the grain boundaries of the bismuth crystals.[1] The data (corrected for decay to an arbitrary zero time of 8-15-51) for all runs are shown in Table I. The temperature of 200°C was chosen since it is anticipated that the surface of bismuth targets in cyclotrons and linear accelerators will reach this temperature. The same sample was used for all runs. Each run (except No. 9-10 in which case the same collecting plate was used for both runs) consisted of placing a new collecting plate in contact with the cooling finger, assembling the apparatus, evacuating, carrying the sample through its temperature cycle, disassembling, and removing the collecting plate from the cooling finger. The quantity of polonium collected on the plate was determined by an ionization-type instrument which was capable of measuring its disintegration rate with an error of ± 5 per cent.

An autoradiograph of each collecting plate was made to determine the efficiency of collection of the polonium. A representative example of such an autoradiograph is given in Figure 1 (Run 12). It can be seen in this figure that essentially all the polonium lost from the one-inch-diameter surface sample condensed directly above it on the collecting plate. The dots of activity surrounding the main portion are the result of leakage from the reverse side of the sample via screw holes. This extraneous activity introduces a relatively small error.

During each run (except No. 7, 8, 9, 13, and 14) the sample was taken through a temperature cycle similar to that given in Figure 2 (Run 12). The temperature on the plateau is designated as temperature, t, in Table I. The length of time at which temperature, t, remained relatively constant (third column from left, Table I) was arbitrarily used as the basis to calculate the loss per hour (fourth and fifth columns from left, Table I). In Runs 7, 8, and 9 the plateau temperature did not remain relatively constant but varied gradually (as a result of experimental difficulties) between the lower and upper temperatures indicated in Table I. Runs 13 and 14 were designed to determine the loss of polonium during

the heating and cooling periods only. The temperature cycle during these runs is shown in Figure 2 (Run 13). The data for these runs (Table I) shows that appreciable loss occurs during the heating and cooling periods which would make the loss per hour for short runs abnormally high when calculated as described above.

Operating pressures for all runs were similar to those indicated for Run 12 in Figure 2.

The comparative efficiencies of dry ice-acetone and tap water as cooling agents for the cooling finger used in the apparatus were determined in two pairs of runs, No. 11 and 12 and No. 13 and 14. Conditions in each of these pairs of runs were identical except for the cooling agent. In both cases the quantity of polonium collected during the water-cooled run was slightly lower (Table I). Autoradiographs made under identical conditions for each pair of collecting plates showed slightly more spreading in the case of the water-cooled plate. This can be seen by comparing the autoradiographs of Runs 11 and 12 shown in Figure 1.

The results of all runs are shown graphically in Figure 3. It is of interest to note that all the points from runs with plateau temperatures near 200°C approach a smooth curve which reaches a constant value after about 30 hours. Run 8, however, which deviates considerably (+ 40°C) from 200°C shows a much higher loss. It also should be pointed out that the total loss for all runs is 5×10^{-7} curie per square centimeter (Table I). This loss is equivalent to losing all the polonium in a 0.13-micron layer of the bismuth used (3.84 millicuries polonium-210 per gram of bismuth) which shows that the percentage loss in yield from a reasonably thick, uncovered, bismuth target would not be appreciable. The contamination which results from this loss may be objectionable, however.

REFERENCES

1. Otto, J. B. and Power, W. H., Rpt. Gen. Res., MLM-597, p. 51, August 7, 1951.

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TABLE I

LOSS OF POLONIUM FROM A BISMUTH-POLONIUM SURFACE*

RUN No.	TOTAL LOSS PER RUN (dis./min./cm.^{2})	TEMPERATURE, t (hr.)	LOSS PER HOUR (dis./min./cm.^{2})	LOSS PER HOUR (C./cm.^{2})	TOTAL TIME AT TEMPERATURE, t (hr.)	TEMPERATURE, t (°C.)	COOLING AGENT
1	134. x 10^3	1	134. x 10^3	60.4 x 10^{-9}	1.0	204-210	DRY ICE-ACETONE
2	211. x 10^3	1.25	169. x 10^3	76.2 x 10^{-9}	2.25	200-205	DRY ICE-ACETONE
3	74.6 x 10^3	1.25	59.6 x 10^3	26.8 x 10^{-9}	3.50	200-207	DRY ICE-ACETONE
4	86.5 x 10^3	3.25	26.6 x 10^3	12.0 x 10^{-9}	6.75	200-204	DRY ICE-ACETONE
5	229. x 10^3	5	45 8 x 10^3	20 6 x 10^{-9}	11.75	200-204	DRY ICE-ACETONE
6	80.7 x 10^3	5	16.1 x 10^3	7.25 x 10^{-9}	16.75	200-201	WATER, 16°C
7	19.4 x 10^3	~ 5	3.88 x 10^3	1.75 x 10^{-9}	21.75	~170-190	DRY ICE-ACETONE
8	160. x 10^3	~ 4.	40. x 10^3	18.0 x 10^{-9}	25.75	204-240	DRY ICE-ACETONE
9-10	47.7 x 10^3	~11.5	4.15 x 10^3	1.87 x 10^{-9}	37.25	9 (6½ HR.) 140-184	DRY ICE-ACETONE
						10 (5 HR.) 202 ± 2	
11	19.3 x 10^3	5.	3.86 x 10^3	1.74 x 10^{-9}	42.25	202 ± 2	WATER, 18.5°C
12	20.5 x 10^3	5.	4.1 x 10^3	1.85 x 10^{-9}	47.25	202 ± 2	DRY ICE-ACETONE
13**	7.17 x 10^3	0.	-	-	-	-	DRY ICE-ACETONE
14**	6.08 x 10^3	0.	-	-	-	-	WATER, 16°C.

1.1 x 10^6 DIS./MIN./CM.^{2} ≡ 5 x 10^{-7} CURIES/CM.^{2} = TOTAL LOSS FROM SAMPLE FOR ALL RUNS.

* THE SAMPLE CONTAINED 3.84 MILLICURIES OF Po^{210} PER GRAM OF BISMUTH.

** HEATING AND COOLING CYCLES ONLY.

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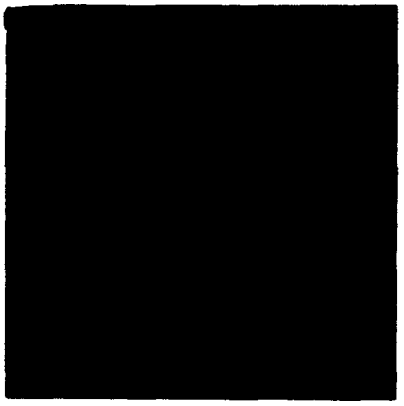
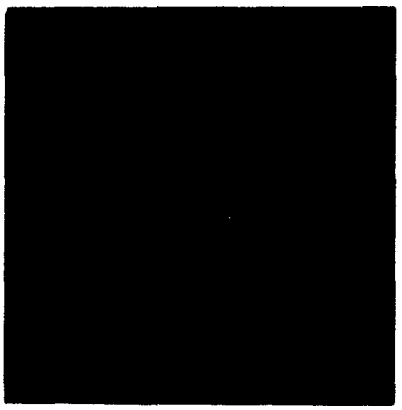


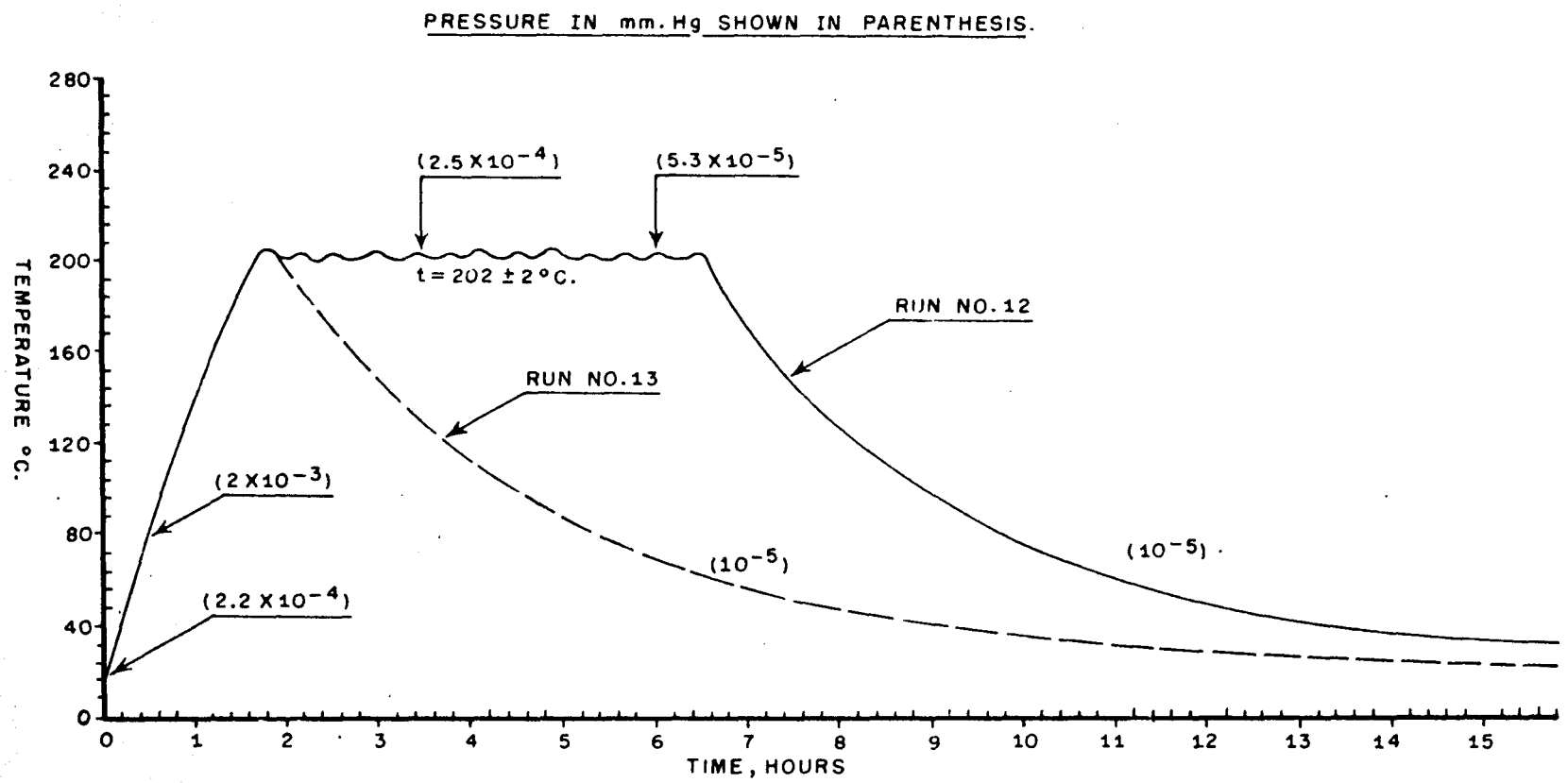
RUN NO. 11 (WATER COOLED)



RUN NO. 12 (DRY ICE-ACETONE COOLED)

EXPOSURE FOR BOTH RUNS: 65 HOURS

AUTORADIOGRAPHS OF COLLECTION
PLATES FROM RUNS 11 AND 12



TEMPERATURE CYCLES USED IN THE STUDY OF THE LOSS OF Po FROM A Po-Bi SURFACE.

FIGURE 2

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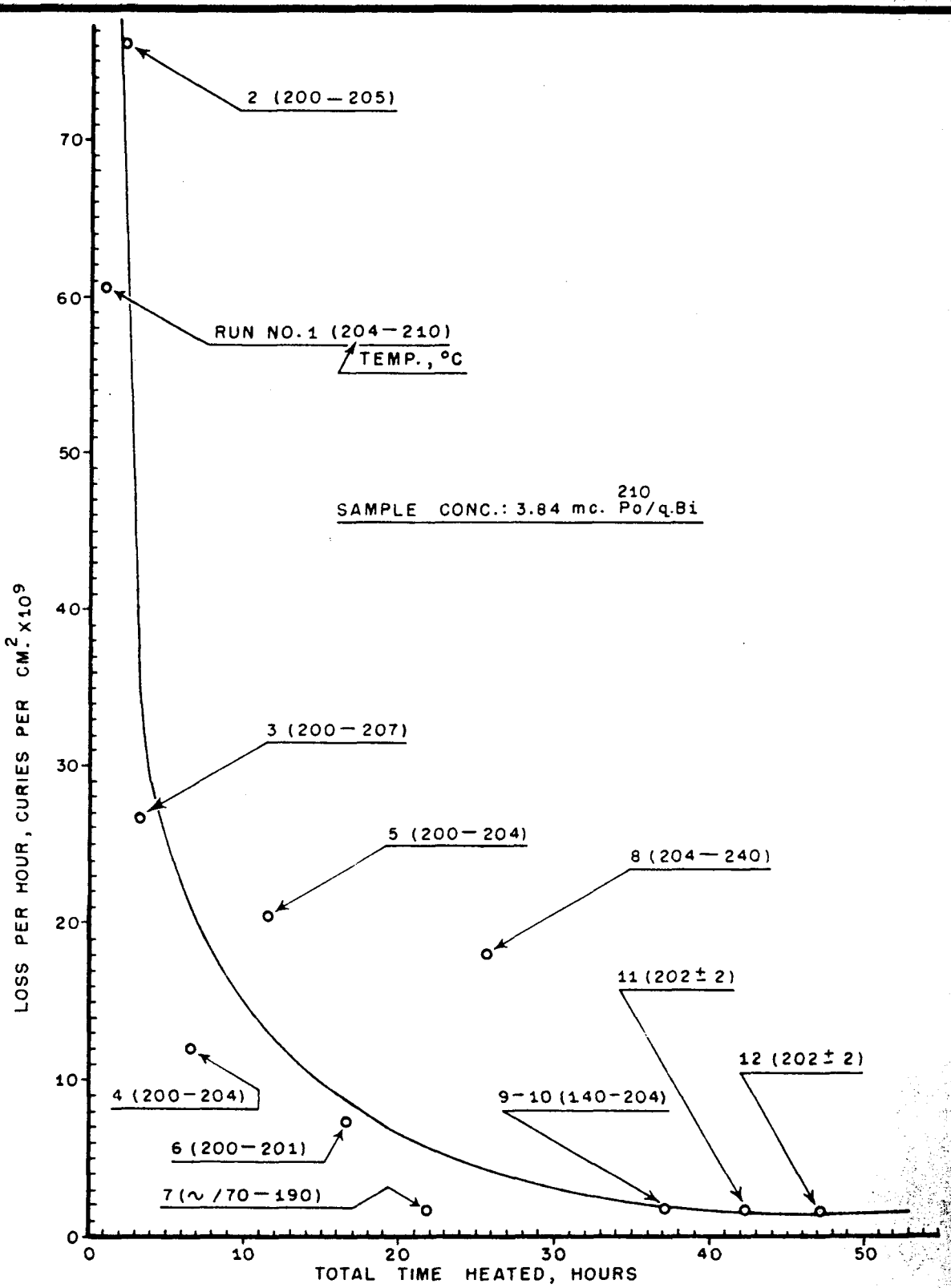
UNCLASSIFIED



VARIATION IN THE LOSS OF Po FROM A Bi—Po SURFACE WITH HEATING TIME.

5106

Problem Title -- **Decontamination of Copper**

Report By -- **W. C. McCluggage**

Work Done By -- **W. C. McCluggage**

INTRODUCTION

During the proton bombardment of bismuth targets in the Oak Ridge cyclotron, the copper dees become contaminated with polonium. Periodically, these dees must be removed and decontaminated before they can be repaired or used with some other target material. Mechanical methods of scouring, scrubbing, and polishing, generally used for polonium removal from metal, are undesirable according to Oak Ridge[1] because of personnel exposure.

DETAILED REPORT

A dipping procedure is being sought capable of reducing the contamination from 2,000 counts per minute per square centimeter to values approaching zero. The inorganic acids and some organic complexing agents are being considered. Electrocleaning and brightening methods will also be studied. The spontaneous plating of polonium onto copper is a major obstacle to easy removal of activity. Mild anodizing of the copper dees will be investigated as a means of preventing this spontaneous deposition.

Preliminary work has been started on this problem.

REFERENCE

1. Case, N., Personal communication.

Problem Title — **Mass Spectrograph**

Report By — **D. B. Wehmeyer**

Work Done By — **W. S. Coakley, J. F. Combs, D. L. Haas, and D. B. Wehmeyer**

INTRODUCTION

A double-focusing mass spectrograph for the analysis of solids has been developed previously.[1] This spectrograph has been assigned to the problem of determining the relative abundance of the 208 and 209 isotopes in the polonium produced in the Oak Ridge cyclotron by proton bombardment of bismuth. In order to make this analysis with the mass spectrograph, several factors must first be investigated. Ion-source studies are being made to find a source which will be efficient in producing ions using small quantities of polonium. The spectrograph is being operated in the high mass range to determine optimum operating constants. Runs are being made with other materials to determine source stability and to help design better monitoring equipment for the ion beam. Electrometer detection of the separated ion beam has been designed to replace photographic-plate detection for accurate isotopic-abundance measurements. The investigation of these problems has been complicated by operational difficulties.

DETAILED REPORT

The investigation of any new material in a mass spectrograph requires considerable preliminary work before a final run can be made. The problem of determining the relative abundance of polonium isotopes is further complicated because the material in the spectrograph will constitute a health hazard. Thus, preliminary studies on polonium are being conducted in a sealed hood, while operation tests on the spectrograph are being made with substitute materials. It is planned that only one run to determine the relative abundance of polonium-208 and -209 will be made in the spectrograph. Because of this, good results may not be obtainable.

Source

Before a mass spectrum of polonium isotopes can be measured, a means of ionization must be found that will produce a measurable number of polonium ions from a 10-microgram sample. Ionization of this small a sample of other materials is feasible with hot-filament and spark sources. These sources may be inoperable using small samples of polonium because of its high vapor pressure. Electron bombardment and Shaw-type[2] sources, usually used with larger samples, may prove useful in modified form.

A study has been completed with the hot-filament type of source. There appears to be no appreciable ionization of polonium by this method. Considerable success has been obtained with this source for other materials, so it merits some discussion. Other experimenters[3] have placed small samples of a material on a tungsten filament which is then heated to 1,000° to 2,000°C. Positive ions are formed if the substance placed on the filament has a lower ionization potential than the work function of tungsten, 4.52 volts (or tungsten oxide, 9.2 volts, if the filament is pretreated). Most of the materials satisfying these

conditions are alkali metals, alkaline earths, or rare earths. Good results have been obtained with radioactive fission products in the rare-earth region using this source. The ionization potential of polonium is not known, but estimates indicate that it is high. An extrapolated value of 8 volts has been obtained by plotting ionization potentials of the other members of this group.

Since surface ionization is an ideal method for producing ions from small samples, it was felt that it should be tried. The experimental apparatus has been described previously.[1] A tungsten-ribbon filament, 0.030-inch wide and 0.001-inch thick, was coated with samples of polonium ranging from 0.002 to 0.5 microgram. Two probes were placed at equal distances from the filament, one probe at filament potential and the other at -1,000 volts. The filament was heated to temperatures from 1 000°C to over 2,500°C. The filament-potential probe would collect only neutral molecules, while the high-potential probe would collect both neutral molecules and positively-charged ions. After each run the probes were examined for alpha activity and the results compared. Ten runs were made, and no ion currents greater than 10^{-13} ampere were found. These results indicate that polonium was not ionized sufficiently by surface ionization to permit the use of this source in the spectrograph where an ion current of an estimated 10^{-11} ampere is needed.

A similar experimental arrangement has been built with a Shaw-type source in place of the hot filament. This source may give better results since some of the ionization obtained is apparently due to electron bombardment. Preliminary tests are being made on this source, and preparations are being made for operation in a hood using a polonium sample in the crucible.

Stability of the Spectrograph

Health hazards may make it impossible to make more than one run with a polonium sample with the spectrograph in its present location. Hence, when the run is made, it will be important to have stable and reproducible operation of all the spectrograph components. To test the stability and reproducibility several runs were made in the spectrograph using lead in a Shaw-type source. Lead was used as a substitute for polonium because its mass, vapor pressure, ionization potential, and place in the periodic table are close to those of polonium. A number of photographic plates were taken, but the spectra observed on the plates did not always correspond with the power input for the source or with the probe readings of the ion current, and in general were not reproducible.

Source-reproducibility studies have been made with lanthanum oxide in the Shaw-source crucible. Measurements of the ion current produced were made by insulating the first slit of the spectrograph and using it as a probe. Ion current to the slit was approximately 2 microamperes and could be read directly on a meter without amplification. The ion-current readings were reproducible and fairly stable. It was found that alignment of the source and slit system was critical, but the ion beam was easily focused on the slits by making mechanical adjustments while watching the meter. Rough alignment of the source-slit geometry was possible with the use of two cathetometers. Although correlation of source constants with slit current is now established, there remains the further correlation with the photographic plate. Studies to establish this correlation are now in progress. The slit has been kept as a monitor of ion current, since readings on a probe farther from the source have not been reproducible or stable.

Detection

The density of a line produced on a photographic plate by an ion beam is not directly proportional to the number of ions which hit the plate. For this reason, electrometer rather than photographic techniques are generally used for very accurate measurements of isotopic abundance. However, for moderately accurate measurements photographic plates can be calibrated with known isotopic ratios, and thus serve as a fairly easy method. For the first measurements of the isotopic abundance of polonium-208 and -209, it is planned to use the photographic method. Therefore preliminary alignment, etc., will be done with photographic plates. At a later time greater accuracy may be needed, so the Electronics Section of this Laboratory has designed a dual set of direct current amplifiers for electrometer detection. It is planned that two probes will be used, one to monitor the entire ion beam, and the other to measure the current attributed to an individual isotope. The electrometers and amplifiers that have been designed will be able to detect 10^{-14} ampere and will be very stable in operation.

Operational Difficulties

The analyzer tube was completely disassembled in July, 1951 to build a new tube for spectrometer work. When the spectrograph was reassembled, a great many vacuum leaks appeared. The entire disassembly procedure had to be gone through several times before the vacuum system became reasonably tight. When the pole pieces were apart, they were nickel plated to prevent oxidation which would cause inhomogeneities in the magnetic field.

Several electrometers used to measure probe currents less than 10^{-8} ampere at some distance from the source have been unstable due to stray electric and magnetic fields. Electric and magnetic shielding of the electrometers has not yet proved satisfactory. Converting the first slit into a probe eliminated the need of other probes for source-to-ion-beam correlations but additional probes may be necessary to facilitate correlation with the photographic plate.

SUMMARY

Ion-source studies with polonium-210 in a hood indicate that surface ionization will not work. Study of a Shaw source is continuing.

Stability and reproducibility studies in the spectrograph indicate that the Shaw source is fairly stable and that source-to-ion-beam correlations are reproducible. Source-to-photographic-plate correlations will be made.

The spectrograph will be used to make the first abundance measurements. The spectrometer will be used later for more accuracy. Amplifiers have been designed for the spectrometer.

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