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THE PREPARATION OF PLUTONIUM METAL ON THE ONE-GRAM SCALE BY MEANS OF THE GRAPHITE CENTRIFUGE

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ABSTRACT

The graphite-centrifuge method was investigated as a possible means of reducing plutonium compounds to metal. Satisfactory reductions up to the one-gram scale with excellent yields of metal were obtained. Preliminary work with uranium as a stand-in for plutonium proved unsatisfactory from the standpoint of reduction of plutonium, although excellent methods were developed for the reduction of uranium.

This report covers studies made of the types of halides of uranium and plutonium, of various reducing agents and their effects on reduction and quality of metal produced, and of time-temperature conditions that were most suitable. Approximately 300 reductions were made with the graphite centrifuge to study these factors and to develop the technique.

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THE PREPARATION OF PLUTONIUM METAL ON THE ONE-GRAM SCALE BY MEANS OF THE GRAPHITE CENTRIFUGE

During the development of this project, quantities of Pu^{239} became available in amounts varying between micrograms in the beginning to grams at present, and with anticipated quantities of hundreds of grams for production. Since the plutonium was desired in the metallic state, research was begun on the problem of preparing coherent slugs of metal on the one-gram scale using uranium as a stand-in. This report concerns the work which led to the preparation of one-gram quantities of plutonium by means of the graphite centrifuge.

The Use of Centrifugal Force to Aid in the Collection of Metal on a Small Scale

The reduction of uranium or plutonium halides by alkali or alkaline earth metals is of the thermite type. It is well known that the yield in a single large mass, and the quality of metal produced in such reactions, improve as the scale of operation is increased. The reasons for this improvement are that on the larger scale surface-to-volume ratio is less for the metal product, and the balance of the heat capacity of charge and product vs. that of liner and bomb is more favorable for good yields. Information obtained from the literature and preliminary experiments on the one-gram-scale preparation of uranium showed immediately that it was relatively easy to prepare finely divided metal, but very difficult to obtain the metal in the form of well consolidated buttons. One method which succeeded in causing the reduced metal to form in a consolidated mass was that which employed centrifugal force to throw down the molten metal into the tip of a cone during the reduction. This was accomplished by placing the reaction mixture in a cone-shaped refractory liner which was sealed inside a steel bomb. The bomb was then placed

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in an all-graphite centrifuge which was heated rapidly to a high temperature while rotating. As the reduction took place, the metal was thrown together in the tip of the refractory liner, thus producing a good yield of coherent metal. Application of this technique has given successful reductions even on the 50-milligram scale.

Description and Operation of the Graphite Centrifuge

The apparatus consisted of a graphite rotor, six inches in diameter, rotated inside a high-frequency coil by means of a modified drill-press assembly. The rotor was constructed with four slots, 90° apart to hold the steel bombs which contained the refractory liner with reactants; four reductions could be made simultaneously. When less than four reductions were made at one time, the rotor was balanced with "dummy" bombs. The loaded bombs were packed into the rotor with MgO, which prevented attack on the stee at high temperatures by the graphite.

Fig. 1 shows the manner of assembly of the bomb and rotor. The charge of halide plus reducing agent, shown on the paper at the left, was placed into the cone-shaped crucible of BeO with the halide on top covering the reducing agent. The crucible, after having been covered with a lid of MgO, was placed inside the steel bomb which was then sealed by welding. After the bombs had been packed tightly in the graphite rotor with MgO, the loaded rotor was placed inside a high-frequency coil. Rotary motion from the drill press was conveyed to the rotor by a slot-and-pin connector device. This can be seen in Figs. 2 and 3.

Rapid induction heating of the rotor was obtained from a 50 KW, 3000 cycle, 400 volt generator (Westinghouse). The rotor was rotated at a speed of 900 rpm which developed a centrifugal force about 50 times that of gravity. In the

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early experiments on uranium the temperature of the rotor was raised to 1300-1400° C in about five minutes and this temperature was maintained for five to ten minutes. Later it was found better to maintain the high temperature for only one or two minutes. For plutonium a temperature of 1100° C for three minutes proved most satisfactory. After shutting off the generator, rotation was continued until the temperature of the rotor reached at least 400-500° C. The bombs, after being cooled to room temperature, were sawed open at the top and the contents removed. The amount of slag covering the metal depended upon the porosity of the refractory liner. Fig. 4 is a longitudinal cross section of a bomb which has been fired in the graphite centrifuge. This particular specimen is far from the best, but it clearly shows the layer of slag on top of the button of uranium metal, which is located in the tip of the crucible. Also to be observed in this specimen are the particles of metal (black spongy deposit) clinging to the upper part of the cone. When this occurred, low yields were obtained. Not shown in Fig. 4 is the second auxiliary or retainer lid noted in Fig. 3. For reductions of $PuCl_3$ it was necessary to interpose a lid of NaCl between the MgO and the charge in order to prevent metal from sticking to the lid of MgO. This often happened in the reductions of $PuCl_3$, in which the metal was violently thrown about at the time of the reaction. The lid of NaCl was solid at the time of the reaction, but melted as the temperature of the rotor was raised; any metal which was thrown up to the lid of NaCl fell back into the crucible and combined with the major portion of Pu when the lid of NaCl melted. Whether or not the slag was absorbed by the walls of the refractory crucible depended upon the nature of the refractory and the chemical composition of the slag. In general, chloride slags were more strongly absorbed than others. The best refractory liners for this work were made of highly vitrified

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BeO. The cone-shaped piece of metal shown on the paper at the right side of Fig. 1 is an example of the product from a typical reduction.

Because of the rotation during an actual run it was impossible to measure the temperature of the inside of the bomb. Therefore, it was essential to know the difference or lag in temperature between the inside of the bomb and the rim of the rotor, the temperature of which could be measured during rotation. Stationary runs were made in which the temperature inside the bomb was observed with an optical pyrometer through a small hole drilled into the center. Table I gives the data obtained which correlated the temperature of the inside of the steel bomb with the temperature of the hottest part (rim) of the rotor.

TABLE I

Relation between Temperature Inside Steel Bomb and Temperature of Graphite Rotor.
(Rotor not Rotating)

Time	Temp. of Hottest Part of Rotor °C	Temp. of Inside Tip of Steel Bomb °C	Remarks
1:52.0	25	25	Generator kept at 38-40 KW until 1:55.75 when it was turned down to 25 KW
53.0	1050	----	
54.0	1200	dark red	
.5	1340	----	
55.0	----	1000	
.25	1400	----	
.50	----	1110	
.75	1460	----	
56.0	----	1225	
.5	1405	----	
57.0	----	1270	
.5	1415	----	
.75	----	1300	

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TABLE I (cont'd)

Time	Temp. of Hottest Part of Rotor °C	Temp. of Inside Tip of Steel Bomb °C	Remarks
58.0	----	1325	
.33	1420	----	
.50	----	1325	
.75	1400	----	
59.0	----	1330	
.33	----	----	Generator turned off. Readings taken very rapidly from here to end of run.
.66	1250	1300	
60.0	1150	1225	
.5	1065	1165	
61.0	1000	1100	
62	950	1000	
63	890	950	

From this data "best" time-temperature conditions for actual operation were decided upon. Two temperatures of the rotor were recorded during a run, (a) the hottest portion of the rim, and (b) the temperature of the body which was taken at approximately the center of the outer surface of the steel bomb. Table II gives data for typical runs of (a) uranium and (b) plutonium.

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TABLE II. Time-Temperature Data for Typical Reductions

(a) Uranium (1.32 g of UF_4, 0.32 g of I_2, 0.50 g of Ca)

Time	Temp. of Rim of Rotor	Temp. of Body of Rotor	Power Supplied KW
11:32	Converter turned on	----	40
32.5	1025	----	39
33	1170	945	38
33.5	1260	1050	38
34	1300	1140	34
34.5	1285	1185	34
35	1295	1200	36
35.5	1350	1250	31
36	1345	1265	31
36.5	1360	1290	31
37	1380	1305	29
37.5	1375	1325	27
38	1380	1320	29
38.5	1395	1350	off

(b) Plutonium (1.45 g $PuCl_3$, 110 mg of Li)

Time	Temp. of Rim of Rotor	Temp. of Body of Rotor	Power Supplied KW
3:24	Converter turned on		40
24.5	870	----	40
25	1135	930	38
25.5	1230	1000	30
26	1230	1080	20
26.5	1250	1100	10
27	1110	1085	20
27.5	1125	1075	22
28	1160	1100	17
28.5	1155	1100	17
29	1155	1100	18
29.5	1160	1120	14
30	1155	1115	14
30.25			off

The Use of the Graphite Centrifuge on the 50-milligram Scale

Three modifications of the above technique were used to carry out reductions on the 50-milligram scale. The first modification employed a similar centrifuge, but one which was about 1/3 the size of that used for the one-gram scale. The speed of rotation, however, was increased to obtain a centrifugal force equal

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to that used on the large scale. This miniature rotor device was soon abandoned in preference to an adaptation of the larger rotor assembly: the one-gram-scale steel bombs were drilled out to receive a smaller steel bomb into which was placed the 50-milligram-scale refractory liner and charge. Thus there resulted a doubly sealed bomb which provided added protection in the early experiments with plutonium when least was known about its behavior.

The third modification, which was the most satisfactory, for carrying our 50-milligram reductions consisted of placing the small charge in the tip of the one-gram-scale cones. Immediately above the charge was placed a small lid which effectively made a small crucible out of the large one. In nearly all reductions of plutonium, either on the 50-milligram or one-gram scale, the air inside the bomb was displaced with argon before the lid was welded on.

The reaction mixture for the one-gram scale consisted of enough halide to produce one gram of metal, plus one of the reducing agents, Ca, Ba, Li, etc. When iodine was used, it was added in the ratio of one mole of I_2 per 3 moles of halide. Enough reducing agent was added to give an over-all excess of about 20 percent by weight.

Results:

A. Uranium and Other Stand-ins

1) Approximately 250 reductions were made using uranium halides and various reducing agents. UF_4 reduced with Ca plus I_2 as a "booster" always produced brittle metal which contained considerable amounts of entrapped slag and a high content of iron. Yields obtained were about 90-104 percent, the 104 percent caused by slag trapped inside the metal. The probable function of the iodine

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"booster" in reductions of UF_4 was to form CaI_2 (MP 575° C) which lowered the melting point of the slag of pure CaF_2 (1360° C).

UF_4 reduced with Li, on the other hand, gave very malleable (indicating good purity) metal with yields of about 94-99 percent and with much lower slag and iron content. When Li was used, it was not necessary to add I_2 "booster" because of the lower melting point of the LiF slag compared to that of CaF_2.

2) UF_3 was successfully reduced with Li.

3) UCl_3 was successfully reduced with Ca, Ba, or Li.

4) A mixture of UF_4 plus MnF_2 was successfully reduced with Li or Ca.

5) A mixture of UCl_3 plus $MnCl_2$ was successfully reduced with Na, Ca, or Li.

In the latter two combinations, about 5 percent by wt. of resulting Mn metal in the alloy was used because the melting point of this alloy is between 700 and 800° C, thus more closely approximating the melting point of Pu (630° C) than that of pure U (1130° C). This permitted the use of lower operating temperatures, thereby better simulating the conditions for the reduction of plutonium. Table III gives a summary of a few of the reductions of uranium.

6) $NdCl_3$ was successfully reduced with Ca.

7) $CeCl_3$ was successfully reduced with Ca.

8) $LaCl_3$ was successfully reduced with Li.

9) MnF_2 was successfully reduced with Li.

The last four reductions were carried out in order to determine the "versatility" of the graphite centrifuge. The centrifuge method is much less sensitive to small variations which may cause complete failure when the stationary bomb method is used. It should be pointed out again that reduction of compounds

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TABLE III. Reduction of Uranium Halides in the Graphite Centrifuge [redacted]

Run	Wt. Metal mg	Compound	Reductant	Refractory	Temp. °C	Time min	Button yield %	Character of button
8a	1000	UF_4	Ca(+I_2)	BeO	1325+	4½	104.5	Metal brittle, but of good appearance.
8d	1000	UF_4	Ca(+I_2)	BeO	1325+	4½	92.5	Metal brittle, but of good appearance.
9a	1000	UF_4	Ca(+I_2)	BeO	1325+	8½	101.5	Metal brittle, but of good appearance.
9b	1000	UF_4	Ca(+I_2)	BeO	1325+	8½	99.5	Considerable slag with metal.
9c	1000	UF_4	Ca(+I_2)	BeO	1325+	8½	103.5	Excellent button.
9d	1000	UF_4	Ca(+I_2)	BeO	1325+	8½	100.2	Brittle button, but of good appearance.
12c	1000	UF_4	Ca(+I_2)	BeO	1325	7½	94.0	Small leak in bomb; metal was good.
12d	1000	UF_4	Ca(+I_2)	BeO	1325	7½	90.5	Large leak in bomb; metal was poor in character.
15a	1000	UF_4	Ca(+I_2)	ThO_2	1325+	6½	90.5	Good button, very well formed.
15c	1000	UF_4	Ca(+I_2)	BeO	1325+	6½	100.8	Excellent button.
21b	1000	UO_2 + UF_4	Ca(+I_2)	BeO	1325	6	101.0	Fairly good button with much dark slag.
28d	1000	UF_4	Li	BeO	1350+	2	99.7	Excellent button, very malleable.
31a	1000	UF_3	Li	BeO	1400	1	99.7	Excellent button, very malleable.
41a	1000	UF_4	Li	BeO	1400	1	97.7	Excellent button, very malleable.
44b	1000	UF_4	Li	BeO-CaO	1400	1½	97.3	Excellent button, very malleable.
44c	1000	UF_4	Li	BeO-CaO	1400	1½	98.8	Excellent button, very malleable.
54a	1000	UCl_3	Ba	BeO	1375	1	96.5	Well formed, but porous metal.
54d	1000	UCl_3	Ca	BeO	1375	1	94.5	Well formed, but dense metal.
55c	1000	UF_4	Ba	BeO	1400-	1½	85.8	Slag not well separated from metal.

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TABLE III. (Continued)

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Run	Wt. Metal mg	Compound	Reductant	Refractory	Temp. °C	Time min	Button yield %	Character of button
62a	1000	UCl_3	Na	BeO	1275+	2	63	Bad button because rotor broke during run.
69b	1000	$UF_4 + MnF_2$	Li	BeO	1000	$3\frac{1}{2}$	91	Good metal, very malleable.
76a	1000	$UF_4 + MnF_2$	Li	BeO-CaO	1100	4	99	Poorly formed button of alloy, brittle.
81b	1000	$UCl_3 + MnCl_2$	Li	BeO	1100	$2\frac{1}{2}$	98	Excellently formed button.
29a	50	UF_4	$Ca(+I_2)$	BeO	1300+	$4\frac{1}{2}$	94	Excellent button
34b	50	UF_4	$Li(+I_2)$	BeO	1400	1	94	Excellent button.

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of uranium and other metals was carried out rather extensively to develop the technique for operating the centrifuge, and because gram quantities of plutonium were not available. When sufficient quantities of plutonium were made available, it was soon learned that uranium was not a satisfactory substitute or stand-in for plutonium. (Baker found that the best stand-in for Pu, from the metallurgical standpoint, was Ce. It was not until Pu was actually used, however, that significant progress was made.)

B. Plutonium

1) 50-milligram-scale reduction:

The first attempts to prepare Pu metal at Site Y were on the 50-milligram scale in the graphite centrifuge. It was first proved with U as a stand-in that reduction on such a scale was possible. Best results were obtained by using Li to reduce either PuF_3 or $PuCl_3$. This scale of reduction proved particularly valuable for testing techniques, and to check the quality of larger amounts of halide before use. It should be emphasized again that the smaller the scale of operation, the more magnified are the inherent difficulties of a reduction of this type. Therefore, in Table IV, a yield greater than 50 percent should be considered excellent, a yield of 97 percent, remarkable.

2) One-gram reductions:

The first one-gram (930 mg) reduction of plutonium in the centrifuge was carried out at a time when the belief still persisted that plutonium was similar in properties to uranium. Therefore high temperatures (1300-1400° C) were employed in the reduction of the PuF_3 with Li, resulting in only a 56 percent yield of metal. Fig. 5 shows this metal button as it came out of the bomb--the first piece of plutonium metal ever produced which was larger than a few micrograms.

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TABLE IV. Centrifuge Method - 50-mg Scale

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Run	Wt. Metal, mg	Compound	Reductant	Refractory	Temp. °C	Time, min	Button Yield %	Character of Button
1333	50	PuF_3	Ca $(+I_2)$	BeO	1300	5	------	Black cokey mass.
1355b	50	PuF_3	Li $(+I_2)$	BeO	1350	3	40	Small button, hardness 94. Micro showed 2 phases. MP about 1050°??
1607	60	$PuCl_3$ (0.46% O_2)	Ca	BeO	1100	1½†	------	No solid metal. No solid metal
1608	50	PuF_4 from PuO_2	Li	BeO	1100	2½†	------	Metal brittle, but of good appearance.
1611b	50	$PuCl_3$ (0.46% O_2)	Li	BeO*	1100	2	94	Malleable. Excellent button, density 15.0.
1621a	50	" "	Li	BeO*	1000	2	------	Black cokey mass.
1625b	50	" "	Li	CaO	1100	2†	------	Black cokey mass.
1625c	50	" "	Sr	BeO*	1100	2†	------	Some bright, brittle metal.
1611a	50	" "	Ba	BeO*	1100	2	------	Black cokey mass.
1625a	50	$PuCl_3$ new	Li	BeO*	1100	2†	70	Bright metal. Yield reduced by rotor failure, density 14.0.
1663b	50	" "	Li	UN	1100	3	------	Black cokey mass.
1663c	50	" "	Ca	UN	1100	3	------	Black cokey mass.
1659c	50	" "	Li	Ta	1100	2½	90(est)	Metal produced, but mostly stuck to crucible.

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TABLE IV (cont'd)

Run	Wt. Metal, mg	Compound	Reductant	Refractory	Temp. °C	Time, min	Button Yield %	Character of Button
1632a	50	PuF_4 from PuO_2	Li	BeO*	1100	1½	86	2 small buttons of good metal.
1662a	50	" "	Li	BeO*	1000	3½	68	Poorly formed button.
1659c	100	" "	Ca $(+I_2)$	BeO*	1100	2½	------	Black cokey mass.
1659b	50	" "	K	BeO*	1100	2½	------	Black cokey mass.

* BeO - 7% porosity

†Rotor broke

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Subsequent information on the melting point, obtained both from experiments here and at Chicago, showed that lower temperatures of reduction were advisable. It was found that reduction could be carried out at 1000° C, but that 1100° C gave best results. Table V lists all of the reductions made in the graphite centrifuge on plutonium on a scale greater than 50 mg. Fig. 6 shows a group of 4 buttons of plutonium metal produced in the graphite centrifuge.

Conclusion:

During the time that reductions were being carried out in the graphite centrifuge, Baker developed the technique for reducing U, and Pu in the stationary bomb on the one-gram scale. Such a method, once proper conditions for the reduction had been determined, was easier to carry out, less time consuming, and in general gave a more pure product. The centrifuge method on the one-gram scale has, however, to date given slightly higher yields. The stationary method has now displaced the graphite centrifuge. Nevertheless, the centrifuge served its purpose at a time when it was needed most.

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TABLE V -- Reduction on 1-gram Scale

Run	Wt. Metal mg	Compound	Reductant	Refractory	Temp. °C	Time min.	Button Yield %	Button Density	Remarks
1466a	930	PuF_4	Li	BeO*	1350°	12 min. at temp.	56	17.7	Slightly malleable button. Pu 97.6%. High in Li, Be, Na, Mg and Fe.
1626a 4/29/44	700	$PuCl_3$	Li	BeO	1100	2^+	19	----	Porous BeO lid. 130 mg. button. Rest stuck to lid as metal.
1633a 5/1/44	870	PuF_4 from PuO_2	Li	BeO	1100	$2\frac{1}{2}^+$	73	19.2(C) 18.3(I)	$BaCl_2$ lid over charge. Good button. M.P. about 805°. Violent gas evolution at about 1050° C.
1660 5/6/44	1250	"	Li	BeO	1100	3^+	84.5	15.6(C) 16.7(I)	LiF lid. Good button.
1663a 5/6/44	895	"	Li	BeO	1100	3^+	93	16.1(C) 17.2(I)	NaCl lid. Excellent button. Was absorbed in UN crucible on remelting.
1669a 5/9/44	1050	$PuCl_3$	Li	BeO	1100	$3\frac{1}{2}^+$	88	16.5(I)	NaCl lid. Good button.
1670 5/9/44	515	"	Ca	BeO	1100	$2\frac{1}{2}^+$	54.5	14.5(C)	NaCl lid. Poor button with cokey mass on top.
1737a	225	$PuCl_3$	Li	BeO	1100		91	----	Excellent button, slag layer on top.
1789a	1205	"	Li	BeO	1100		97.5	18.8	Good button. Same chloride as #1784.

* BeO - 0.7% porosity, others 7% porosity.
(C) Density by capillary method
(I) Density by immersion in bromobenzene

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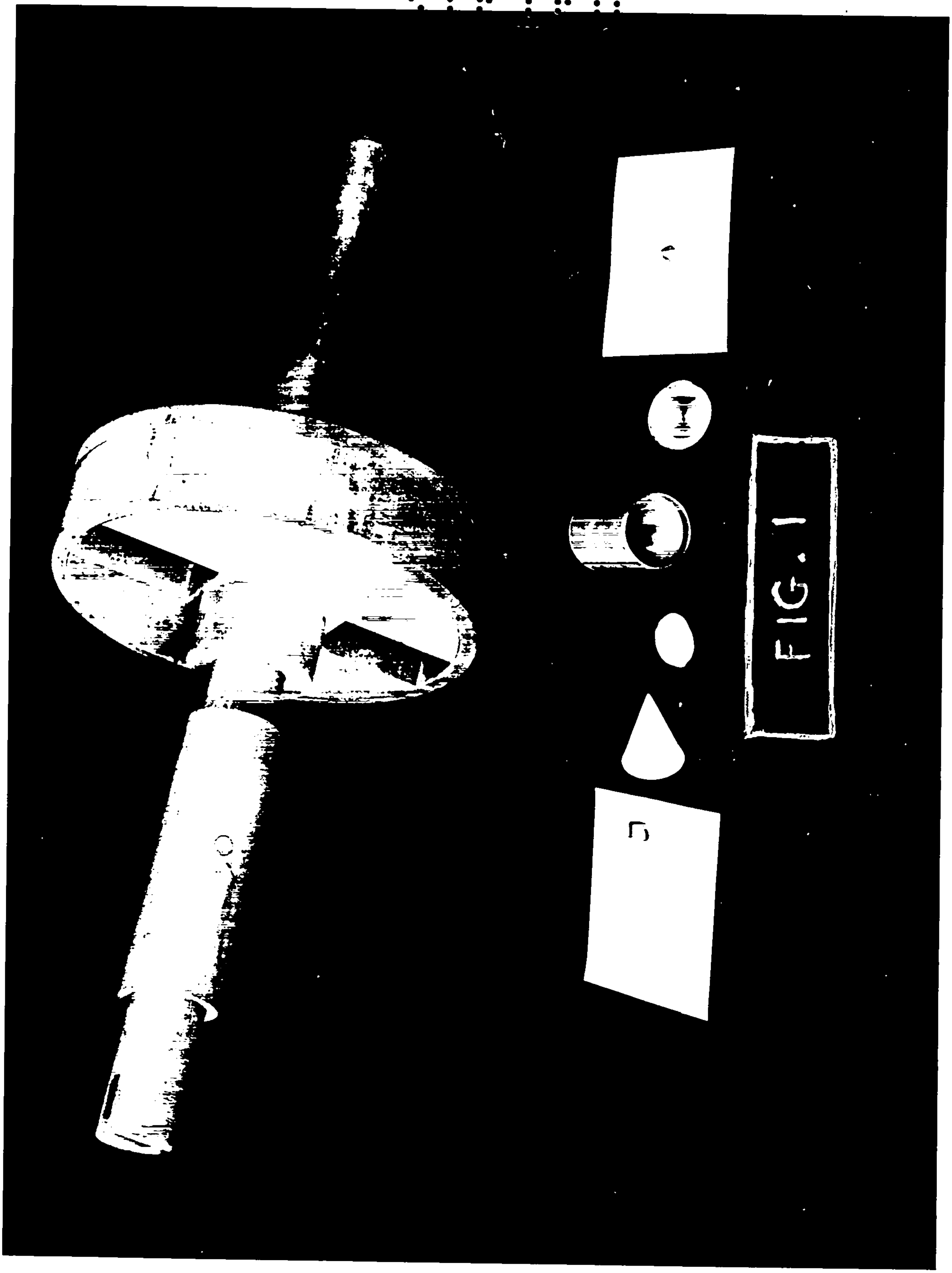

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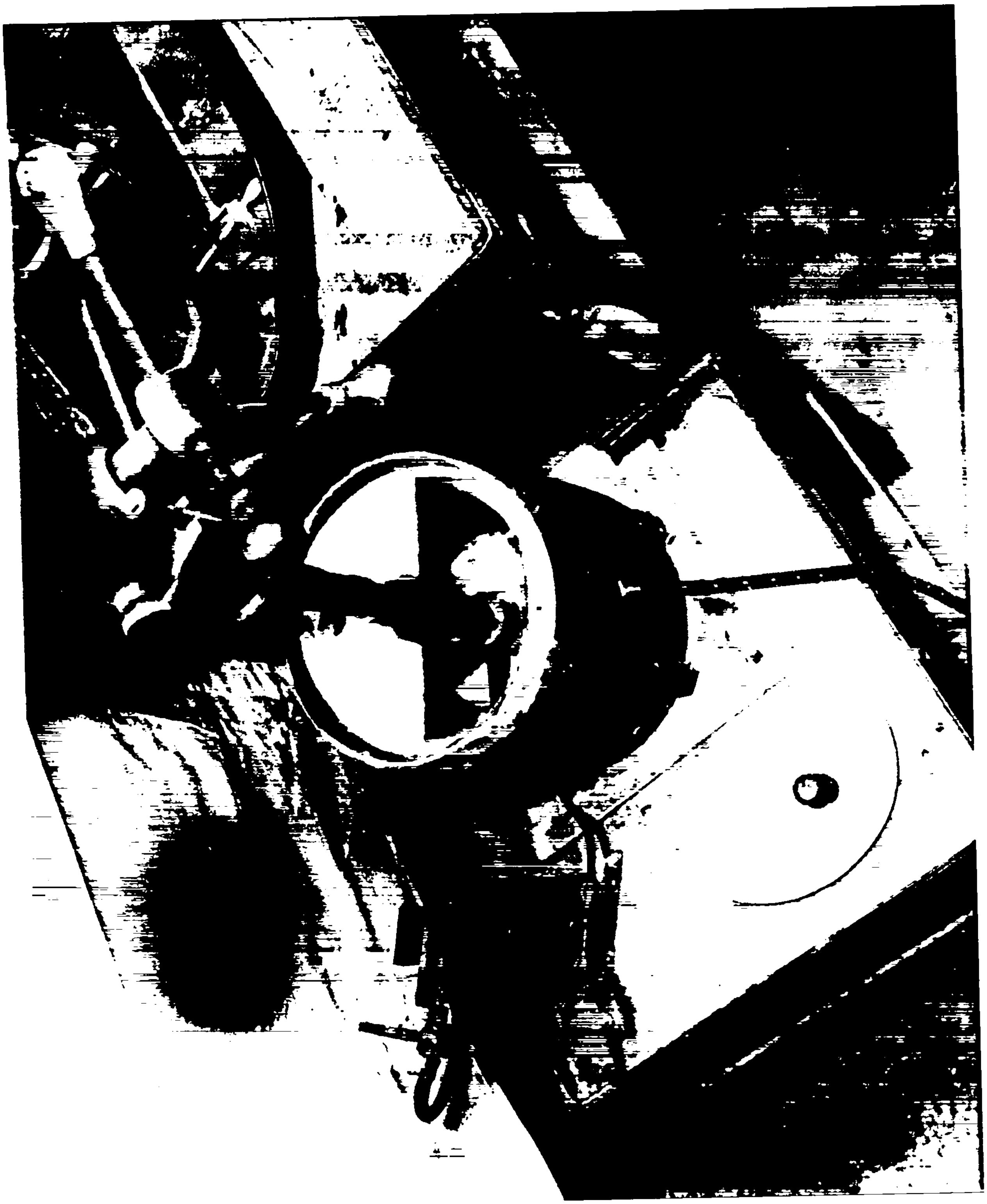

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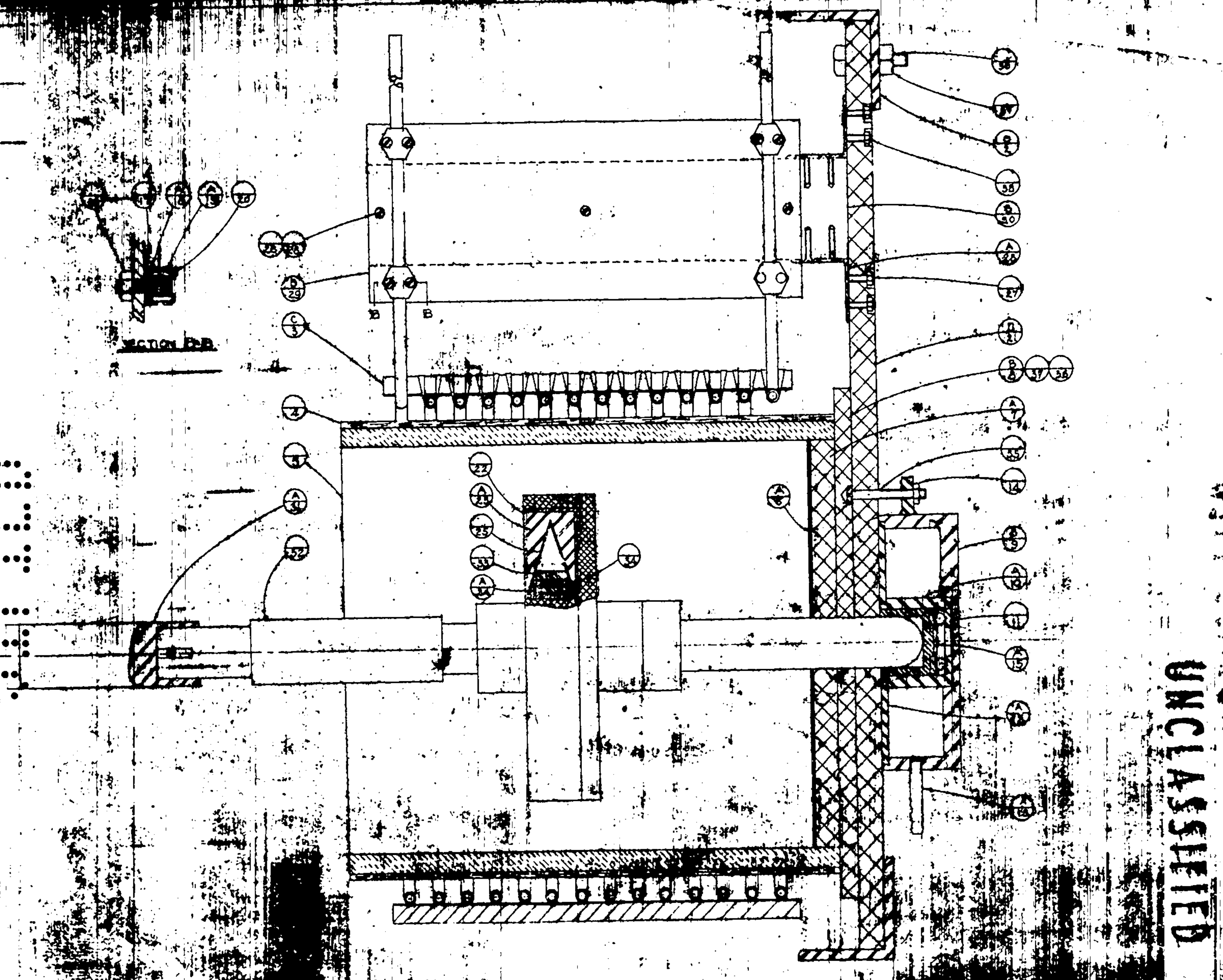

SECTION A-A

FIGURE 3

38	5-40 N.C. HEX NUTS	PURC
37	1/4-20 NC. STD. SQ. NUT	PURC
36	1/4-20 N.C. x 1 1/4 LG. SQ. HD. SCR	PURC
35	10-24 N.C. x 1 1/2 LG. RD. HD. SCR	PURC
34	BERYLLIUM OX. SPACER	
33	BERYLLIUM OXIDE CAP FOR CRUCIBLE	
32	GRAPHITE ROTOR SEE DWG. C-107	
31	DRIVE ADAPTER	DETA
30	POST - FOR COIL SUPPORT PANEL	DETA
29	COIL SUPPORT PANEL	DETA
28	6-32 N.C. x 3/8 LG RD HD MACH. SCR.	PURC
27	5-40 N.C. x 1/4 LG FIL HD MACH. SCR.	PU
26	6-32 N.C. HEX. NUTS	PUR
25	REFRACTORY CRU[illegible]	
24	BOMB COVER	DETA
23	BOMB	DE
22	BERYLLIUM OXIDE PACKING	PURC
21	BASE FOR INDUCTANCE FURNACE	DE
20	5-40 N.C x 1/8 LG. FIL. HD. MACH. SCR.	PU
19	TUBE CLAMP COV[illegible]	DE
18	TUBE CLAMP BRASS	DE
17	[illegible] BRASS WASHER	PUR
16	1/4-20 N.C. HEX NUTS BRASS	PUR
15	SOCKET FOR BEARING RETAINER	DETA
14	No 10-24 N.C. x 1 1/2 LG. RD. HD. MACH. SCR.	PUR
13	WATER HOSE CONNECT	DETA
12	BEARING RETAINER COVER	DET
11	SKF No 2802 THR. BRG.	PUR
10	BEARING RETAINER SLEEVE	DE
9	BEARING RETAINER HOUSING	DE
8	SUB-BASE PLATE	DET
7	INSULATER DISK	DET
6	MICA INSULATER.	DET
5	QUARTZ TUBE 4 I.D. x 10 3/4 LG. [illegible]	PUR
4	ASBESTOS CLOTH 10 3/4 x 30 3/4 LG.	PUR
3	INDUCTANCE COIL	[illegible]
ITEM NO.	DESCRIPTION.	[illegible]

[illegible]E CENTRIFUGE ROTOR AND HIGH [illegible]NCY HEATING UNIT

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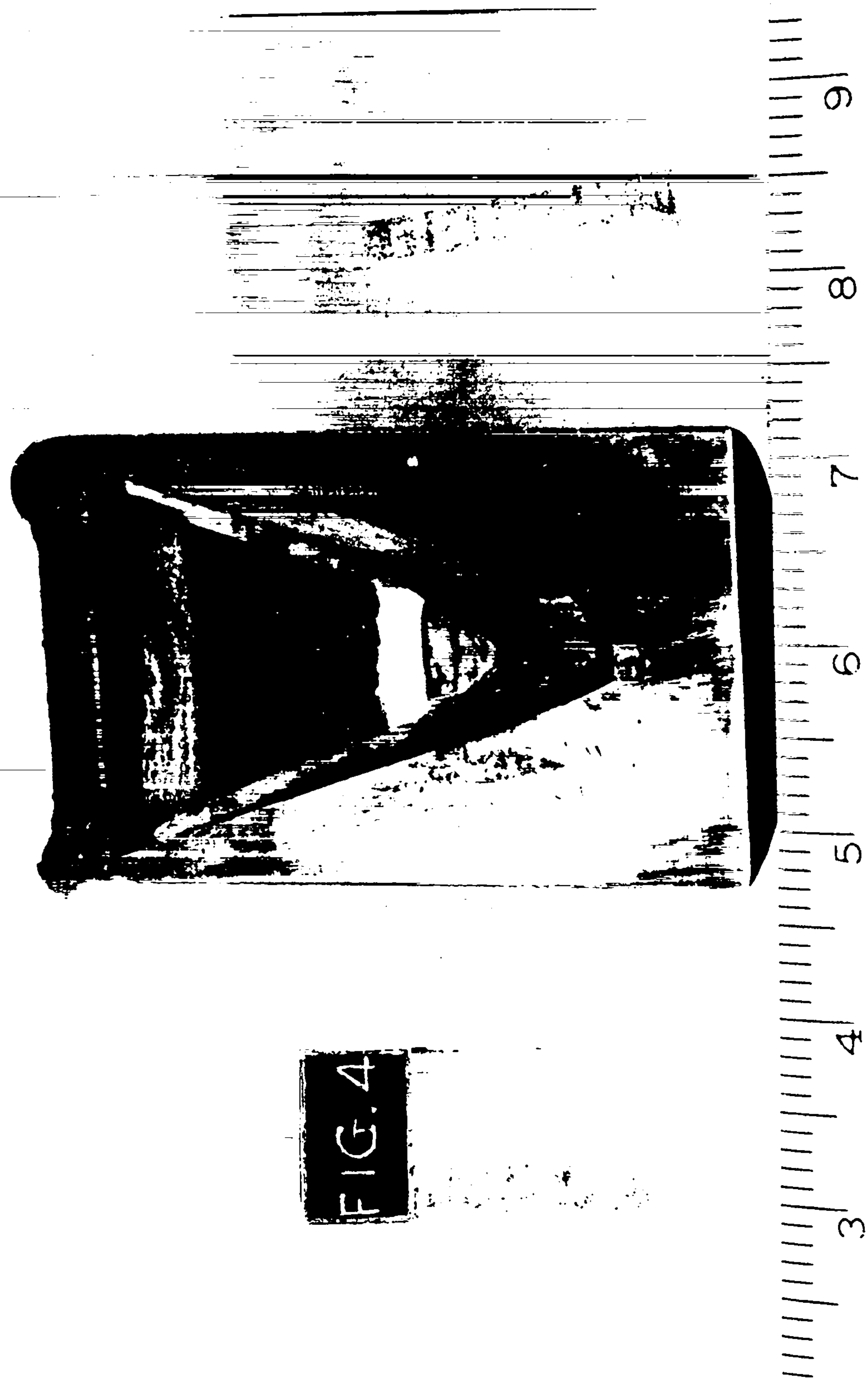

FIG. 4

Fig. 5

Product from First One-gram Reduction of Plutonium
Weight = 520 mg., yield = 56 percent. Sample No. 1466a.

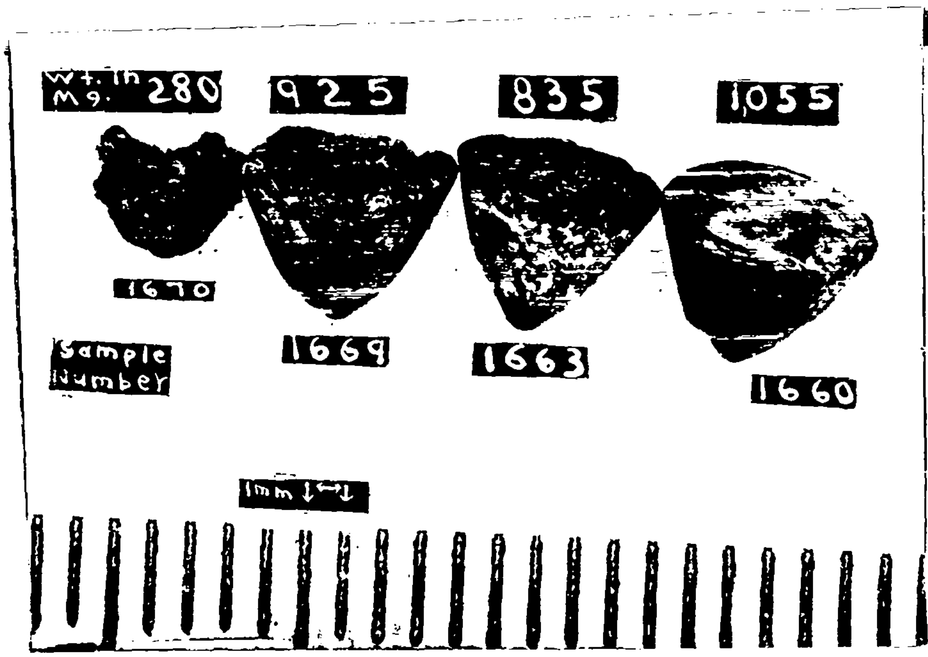

Fig. 6

Product from One-gram Scale Reductions of Plutonium.
Refer to Table V for complete history of buttons.