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APPLICATION OF A NEW TYPE CRUCIBLE TO THE PREPARATION OF URANIUM AND PLUTONIUM METAL BY THE STATIONARY BOMB METHOD

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TECHNOLOGY--PLUTONIUM

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TECHNOLOGY—PLUTONIUM

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ABSTRACT

Magnesium oxide reduction crucibles have been developed that increase the processing efficiency of uranium and plutonium production. The characteristics of the crucibles are described.

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Introduction

The stationary bomb method of preparing high purity uranium and plutonium metal has been in use at Los Alamos for over six years.[2,3] The main reaction involved is the reduction of the tetrafluoride with calcium, using iodine as a booster. This reaction is carried out in an MgO crucible supported in a metal bomb by a layer of granular MgO sand. The metal product forms as a clean, solid, coherent mass with an average yield of 99.75%. The remainder of the metal is distributed non-homogeneously in the MgO crucible, in the process slag, and in the MgO sand. The crucibles or "liners" constitute approximately two-thirds by weight of the total reduction residues.

The crucibles described in this report were developed for the purpose of reducing the weight of MgO sent to recovery. The method of fabrication was developed by A. G. Allison of Group CMR-6.[1] High product purity and good button characteristics have been retained.

Crucible Design

New crucibles have been designed for use in the 500 gm, 1000 gm, and 2000 gm reductions. Drawings of these are shown in Figs. 1, 2, and 3. The curved shape of the crucible bottom is important.

The initial tests of the thin crucibles were made on crucibles which in the unfired state had flat bottoms. On firing, the bottom bowed in-

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ward (toward the inside of the crucible), setting up strains in the crucible bottom which caused the bottom to crack during reduction. By making the bottom curved, its strength was increased, excessive stresses were avoided, and no failures were encountered. Photographs of sectioned and unsectioned crucibles are shown in Figs. 4, 5, 6, and 7. The crucibles shown on the right in Figs. 4 and 5 are the old-type crucibles.

Crucible Preparation

The crucibles are made from fused MgO manufactured by the Norton Company. This raw material has the same purity specifications as the MgO used in the old-type crucible production.

The crucibles are made by first pouring a slurry of MgO into a plaster mold. When the coating on the mold reaches the desired thickness, the excess slurry is poured out, leaving the coating. The coating shrinks slightly on setting and can then be easily removed from the mold. The crucibles are fired at 1750°C in an induction coil.

Details of the slip-casting process are given in LA-1133.[1]

Crucible Testing

All crucible testing was done with normal uranium.

The evaluation of the crucibles was accomplished by using normal uranium tetrafluoride. The charge consisted of UF_4, 0.1 mole I_2/mole U, and 25% excess Ca.

Figures 4, 5, 6, and 7 show the thin-walled crucibles and button in comparison to the former crucibles and buttons. Drawings of the three

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crucibles, 500 gm, 1000 gm, and 2000 gm, with their dimensions, are shown in Figs. 1, 2, and 3. The firing schedules for the three scales of reduction are shown in Tables I, II, and III. The firing schedules may vary slightly, i.e., 15 ± 2 min.

A 20 kw high frequency Ajax convertor is used at Los Alamos as the induction heating unit. All bombs through 1000 gm are fired in the 7 in. I.D. No. 312 Ajax furnace with the bomb arranged coaxially in the coil so that the bottom of the bomb is about 1 in. above the bottom of the coil. The 2 kg bomb extends about 2 in. above the crucible top to prevent failure of the copper gasket. The steel bombs used for the respective crucibles are shown in Figs. 8, 9, and 10. Typical results are shown in Table IV.

The crucible weights vary rather widely. For the 500 gm scale crucible, the weight varies approximately between 450 and 550 gm. The weights of the 1000 and 2000 gm crucibles are about 700 and 1100 gm with variations of about 100 gm and 125 gm, respectively. The metal buttons produced are thinner than the former buttons because of the larger crucible diameter. In some instances this made minor changes necessary in button handling techniques up to and including the fabrication process.

The physical appearance of the buttons has in all cases been excellent. The button bottoms sometimes have rather large indentations which have been shallow and easy to clean. They are caused by blisters

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forming on the inside liner bottom. This minor reaction between the metal button and the crucible bottom is not at all unique with the new type of crucible.

The uranium button yields and purity have shown no change from previous production. The plutonium reduction yields show a small improvement. This may be due to the shorter period of contact between the molten plutonium metal and the liner as a result of the increased rate of cooling associated with the reduced insulation afforded by the thin-walled crucible.

It is necessary to have a slightly larger annular space between the bomb and crucible with the thin-walled crucible, approximately 1/16 in. on diameter. This is due to small differences that may occur in the outside diameter of the crucible. There is a layer, approximately 1/4 in. thick, of MgO packing sand in the bottom of the bomb.

After many tests on reductions made without lids resulting in only minor detrimental effects, the crucible lids have been eliminated. In sporadic instances it was noted that pin-points of uranium metal appeared on the bomb lid. By coating the bomb lid with a thin slurry of MgO (-200 mesh MgO + H_2O), the droplets of uranium were kept from touching the metal of the bomb lid. The coatings are applied the afternoon before the day of the reduction and dried at 100°C overnight. The surfaces to which the coating is applied should be fairly clean. The coating adheres quite well. It is soft but will fall into the charge only under abuse.

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After the reduction, the coating is easily washed off with dilute acetic acid.

The 1000 gm liners were used in production for several months and performed excellently in all respects. The 2 kg liners were put into production on August 19, 1950, using a 2000 gm charge. Performance was and has continued to be excellent. The crucibles in use at the present time are being manufactured by the Norton Company.

Crucible Advantages

The main advantages of the crucibles are:

1. The large decrease in reduction residues.
2. The greater ease in handling throughout the reduction process.
3. For the 2 kg crucible, there are additional advantages in the accountability operations.

The change in reduction residues is tabulated in Table V.

The principal decrease in the MgO content is in the crucible, which is about 70% lighter in weight. The decrease in MgO sand varies up to about 50%, depending on the reduction scale. Considering that a maximum charge of 2000 gm is used, the total decrease in weight of MgO residues averages between 65 to 70%. This results in a reduction of the total solid residues of about 50 to 60%.

To show the comparative ease of handling the fully loaded bombs of different sizes, the bombs and their accompanying weights are shown

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below:

Liner and Bomb Size (gm)	Loaded Bomb Weight (lb)
Former R-2, 1000	32
New A-322A, 1000	23
New A-322, 2000	42
New A-322, 3000	46

The 2 kg crucibles appreciably decrease the work in the reduction process in producing the same quantity of metal product.

It is possible to load the 2000 gm crucible with a 3000 gm charge (providing the charge is tamped into the crucible). This would reduce the comparative MgO weight for the same weight of product to 20% of that of the former method. As shown in Table V, a decrease in total material sent to recovery of 65% would result. The further advantages of the 2000 gm crucible are passed on in the form of easier accountability procedure, less sampling, and fewer analyses.

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REFERENCES

1. A. G. Allison and M. R. Nadler, "Production of Crucibles For Melting and Casting Plutonium and Uranium-235." LA-1133. June 15, 1950.

2. R. D. Baker, "Preparation of Uranium by the Bomb Method." LA-472. June 25, 1946.

3. R. D. Baker, "Preparation of Plutonium by the Bomb Method." LA-473. May 15, 1946.

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Table I

Uranium Firing Schedule for New 500 Gram Crucible

Time (min)	Input to Converter (kw)	Bomb Well Temp. (°C)
0	0-1.5	30
1	4.5	75
2	4.5	105
3	4.0	150
4	4.5	175
5	4.5	220
6	5.5	255
7	6.0	285
8	6.0	325
9	6.0	370
10	6.0	395
11	6.5	425
12	6.5	450
13	6.5	480
14	6.5	595
15	0	695

Fired at: 1345

Power off: 1415

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Table II

Uranium Firing Schedule for New 1000 Gram Crucible

Time (min)	Input to Convertor (kw)	Bomb Well Temp. (°C)
0	0-1.5	30
1	5.0	100
2	5.0	145
3	5.0	180
4	5.0	205
5	5.0	235
6	5.0	265
7	5.5	295
8	5.5	325
9	6.0	370
10	7.5	400
11	7.5	445
12	7.5	485
13	7.5	520
14	7.5	535
15	7.5	640
16	0	700

Fired at: 1445

Power off: 1530

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Table III

Uranium Firing Schedule for New 2000 Gram Crucible

Time (min)	Input to Convertor (kw)	Bomb Well Temp. (°C)
0	0-1.0	30
1	8.5	110
2	8.5	210
3	9.0	310
4	9.0	370
5	8.5	410
6	8.5	445
7	8.5	465
8	8.5	495
9	8.5	520
10	9.0	540
11	11.5	565
12	11.5	600
13	11.5	625
14	11.5	645
15	12.0	675
16	0	745

Fired at: 1525
Power off: 1600

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Table IV

Scale (gm)	Crucible Weight (gm)	Button Yield (%)	Button Purity Analysis	Physical Appearance of Buttons	Liner Performance
500	521	99.65	Meets specifications	Good	Excellent
	578	99.65	"	Good	Excellent
	494	99.74	"	Good	Excellent
	445	99.65	"	Good	Excellent
1000	779	99.90	"	Good	Excellent
	705	99.86	"	Good	Excellent
	628	99.86	"	Good	Excellent
	707	99.64	"	Good	Excellent
2000	1039	99.88	"	Good	Excellent
	1055	99.88	"	Good	Excellent
	1101	99.79	"	Good	Excellent
	1106	99.69	"	Good	Excellent
3000	1006	99.70	"	Good	Excellent
	981	99.67	"	Good	Excellent

Note: "Meets specifications" denotes no change from previous purity.

Good physical appearance of the button and excellent liner performance denotes no change from reductions with other crucibles.

Table V

Scale (gm)	Production				Production With New Crucible				% Less MgO (Liner and Sand)	% Less Total Material Sent To Recovery
	Weight of Liner and Lid (gm)	Weight of MgO Sand (gm)	Weight of Slag (gm)	Total Solid Residue (gm)	Weight of Liner, no Lid (gm)	Weight of MgO Sand (gm)	Weight of Slag (gm)	Total Solid Residue (gm)		
500	1650	270	460	2380	510	200	460	1170	63.0	50.8
1000	2360	735	825	3920	700	385	825	1910	65.0	51.3
2000	4720	1470	1650	7840	1100	650	1650	3400	71.7	56.6
3000	7080	2205	2475	11760	1100	650	2475	4225	81.2	64.1

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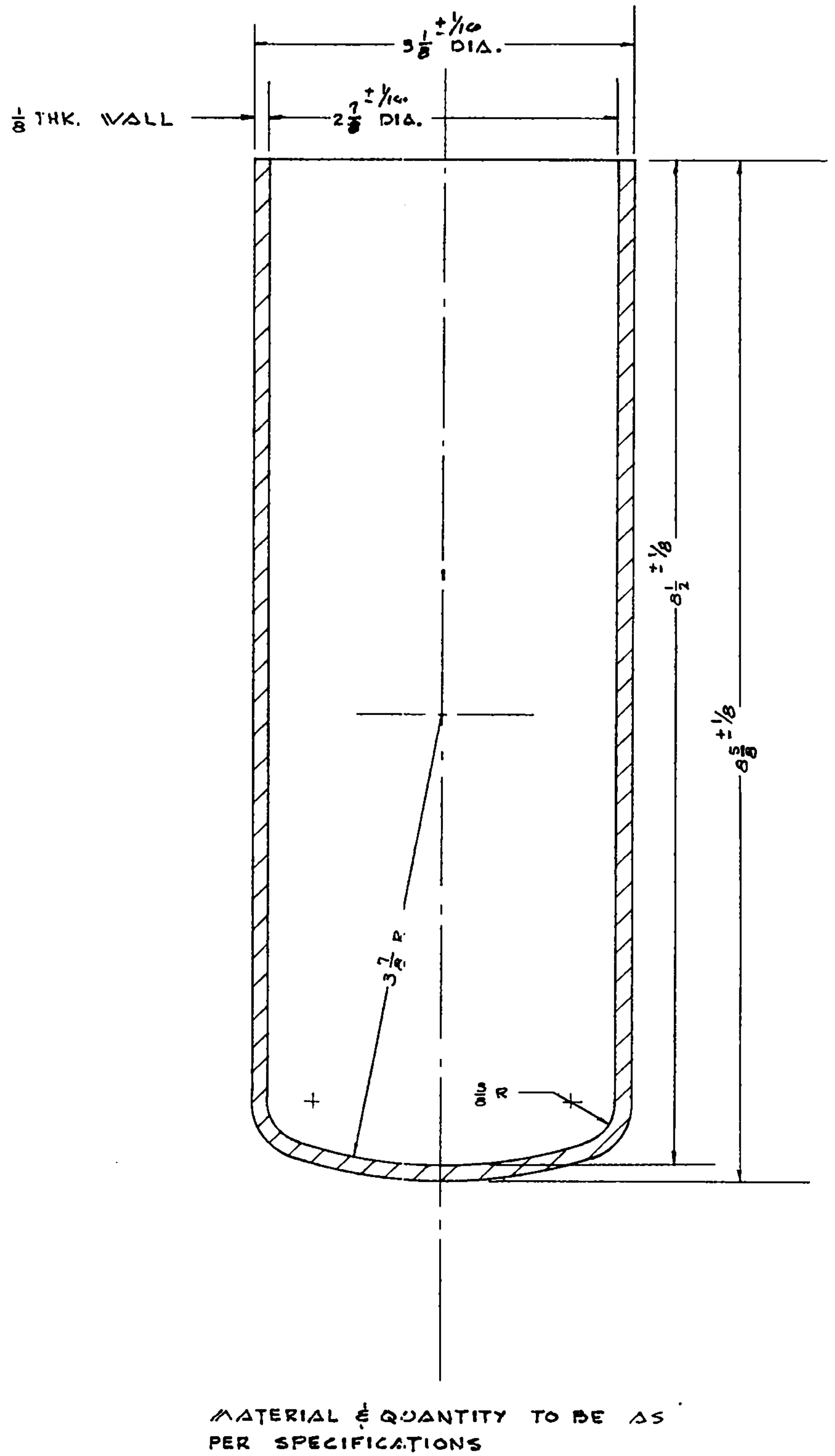

Fig. 1

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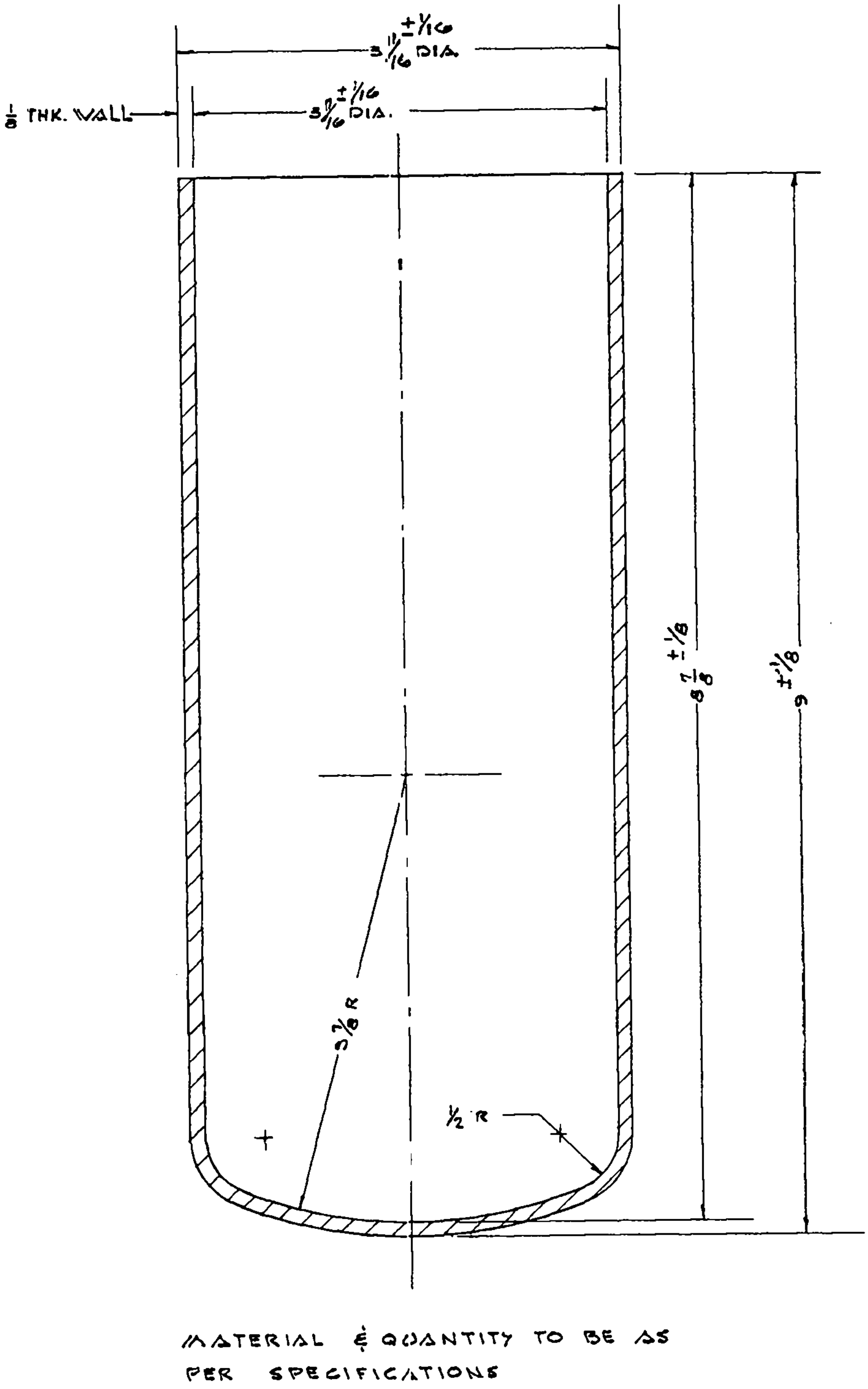

Fig. 2

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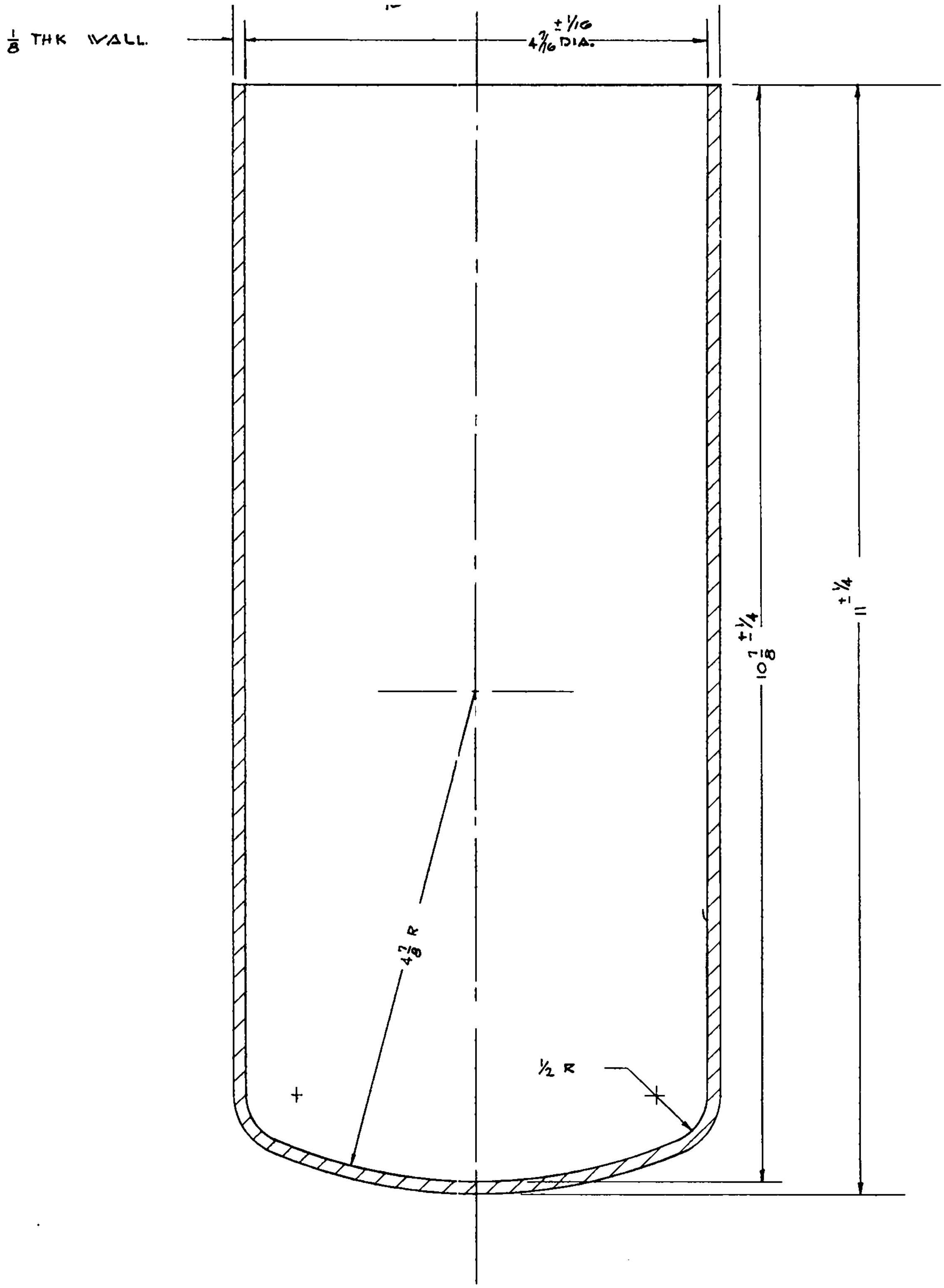

MATERIAL & QUANTITY TO BE AS PER SPECIFICATIONS

Fig. 3

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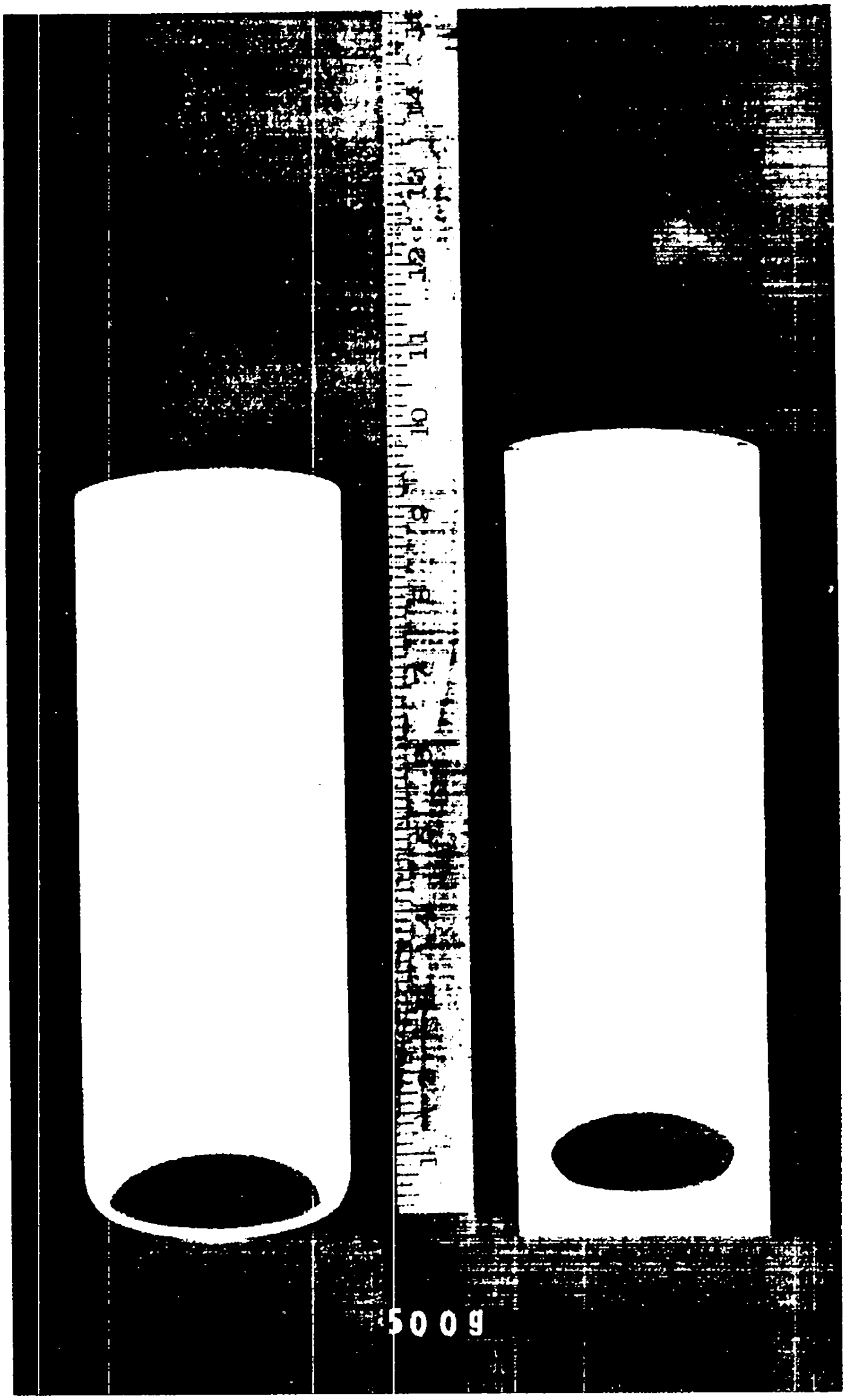

Fig. 4

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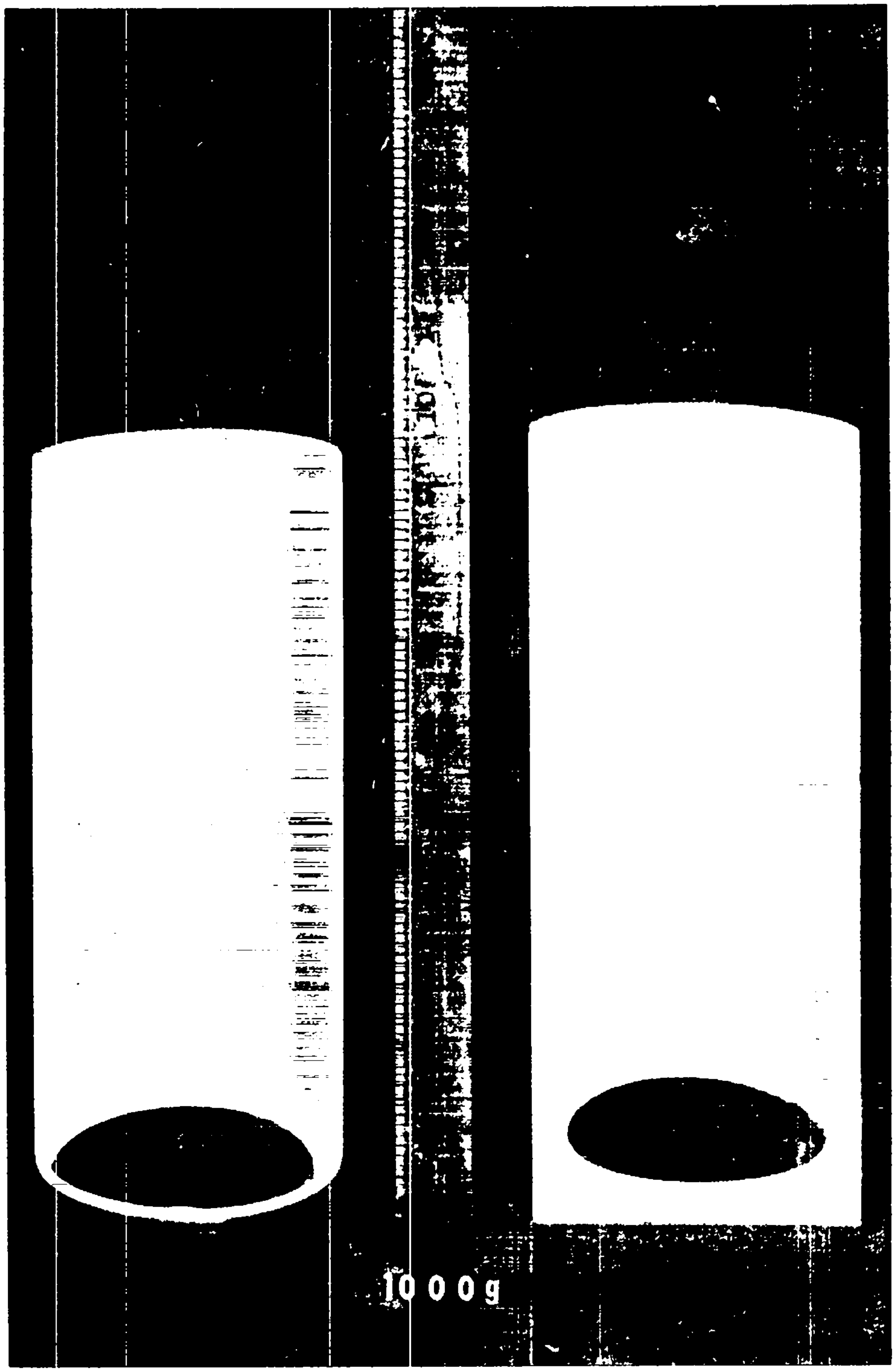

Fig. 5

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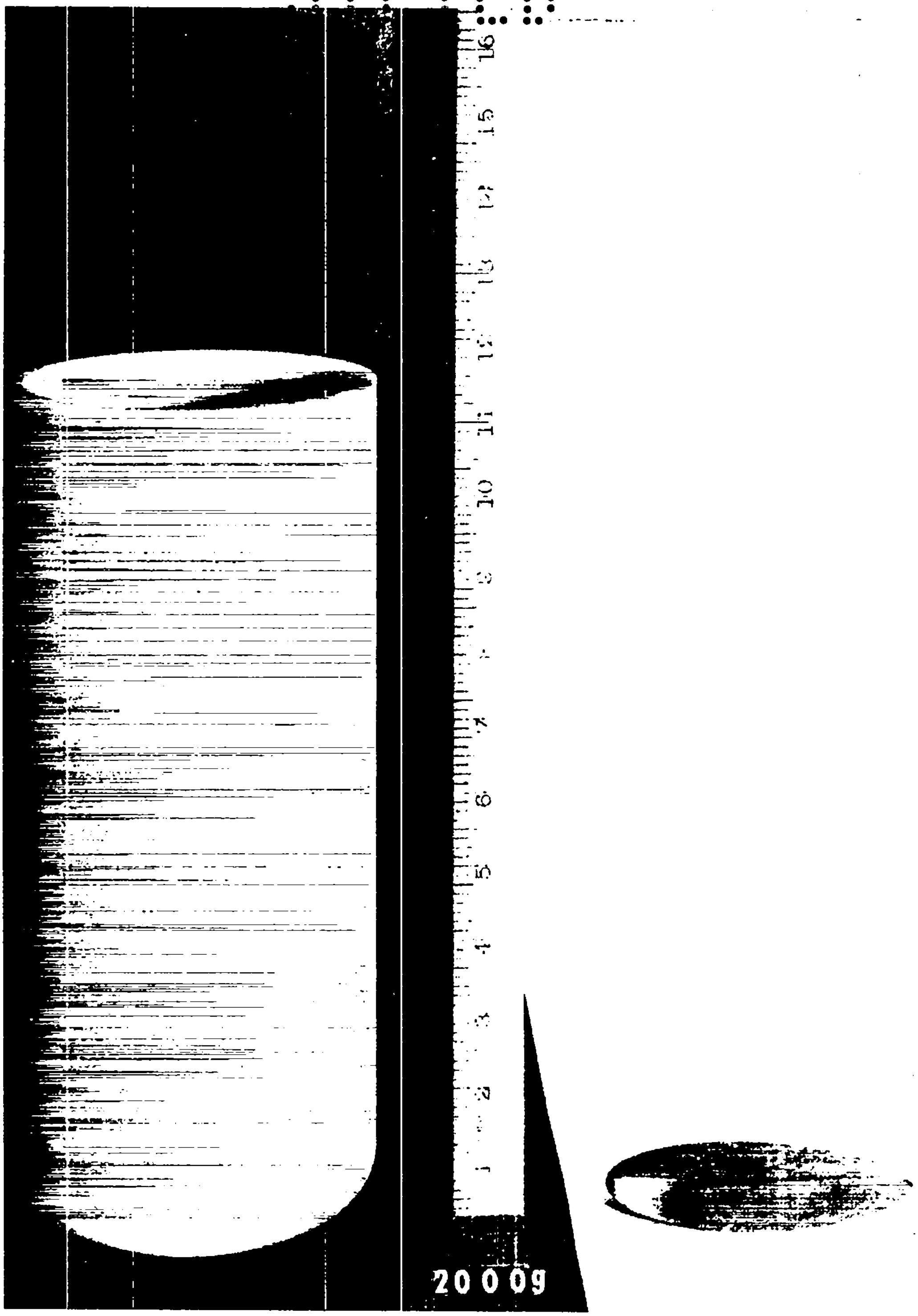

Fig. 6

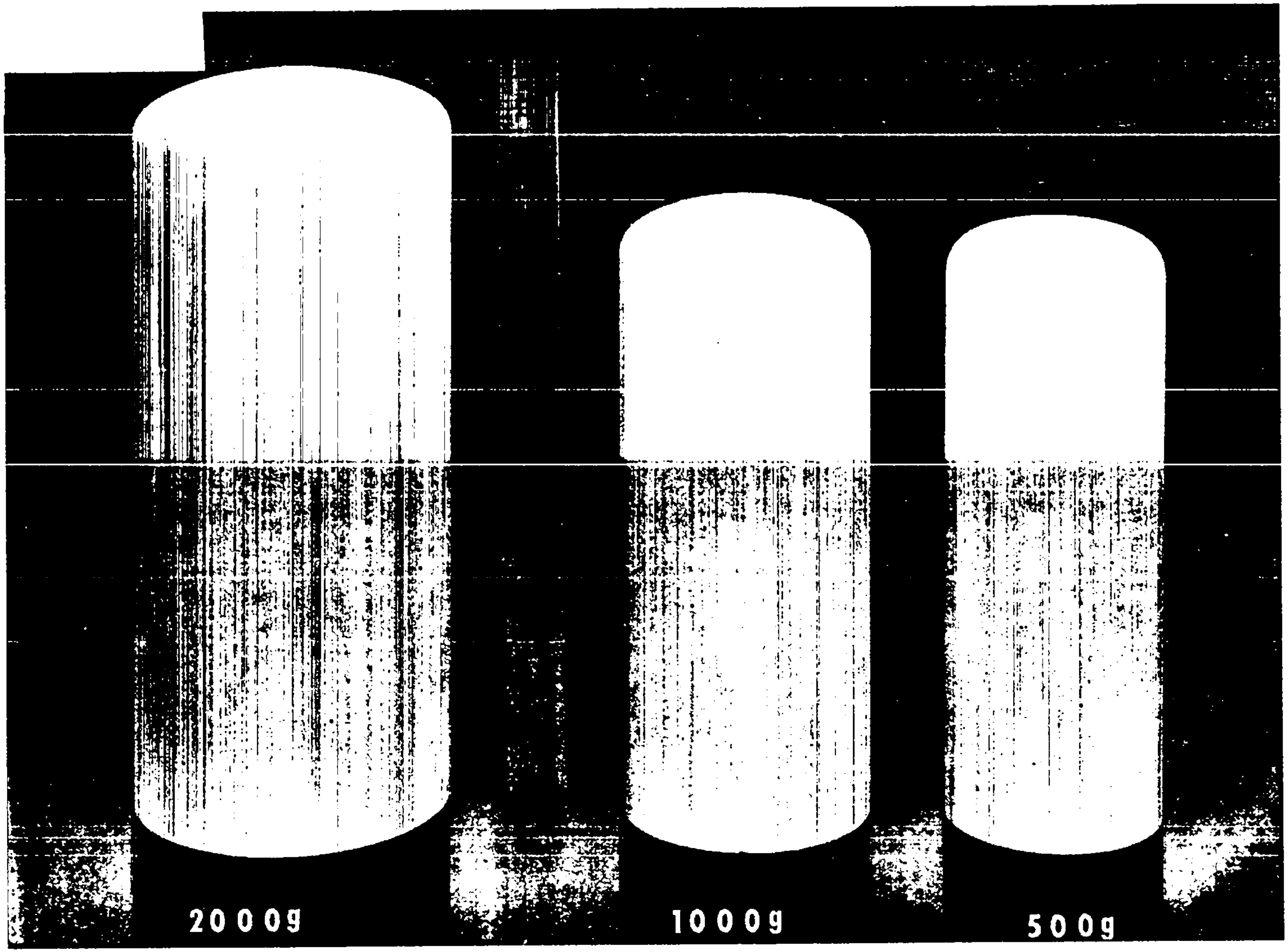

Fig. 7

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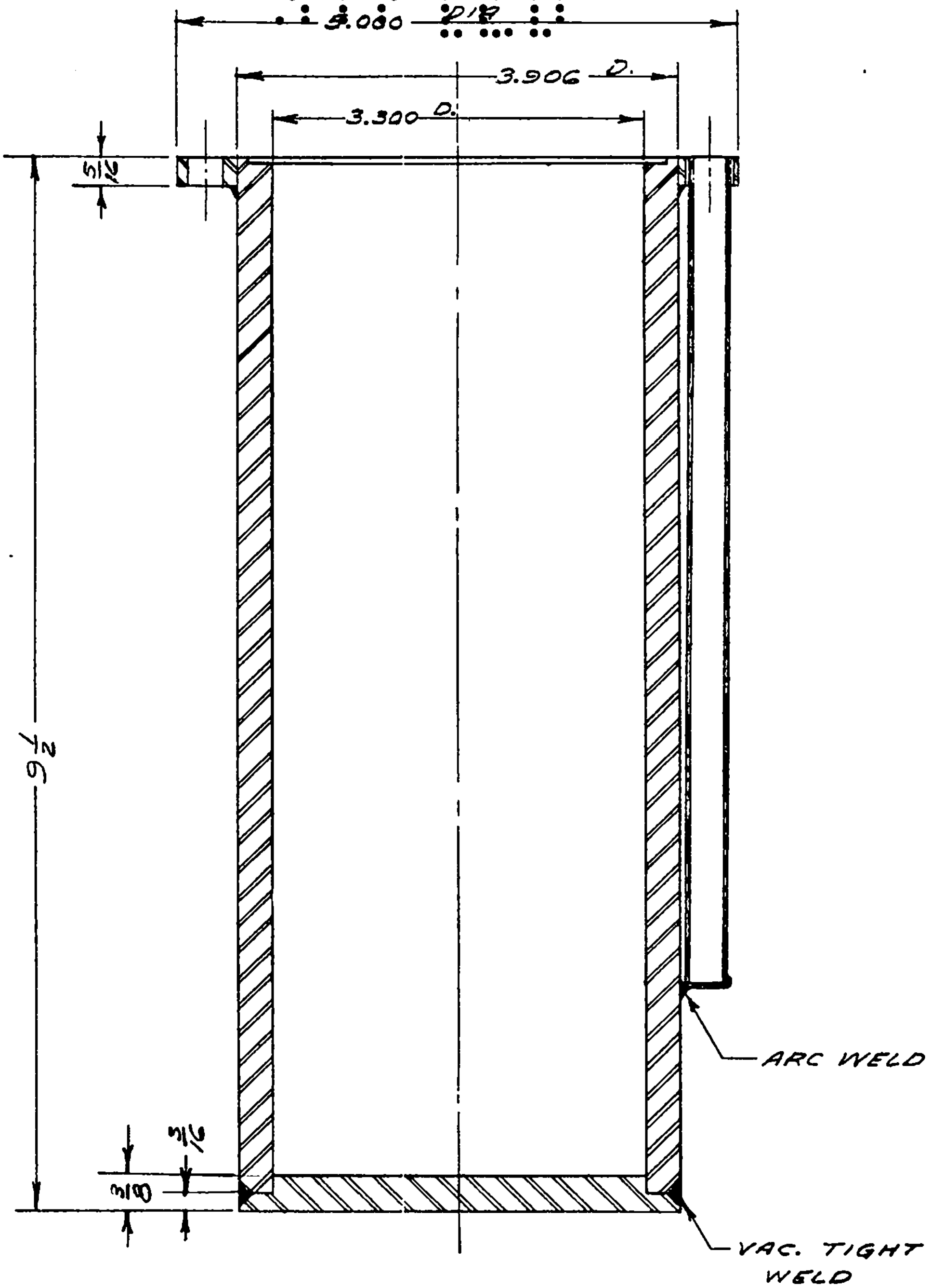

BOMB - 500 GRAM

SCALE 3/4" = 1"

Fig. 8

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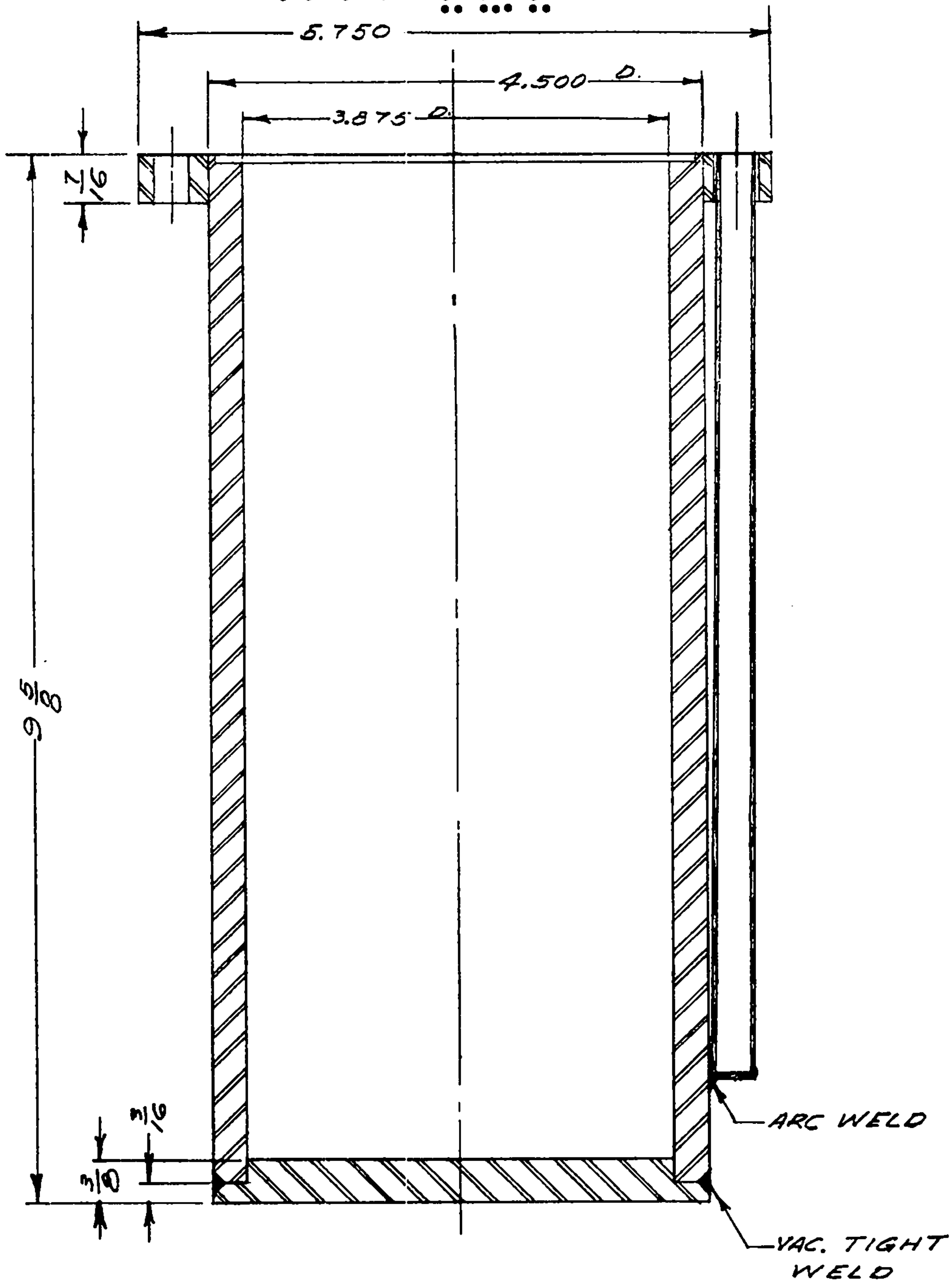

BOMB - 1 KILOGRAM

SCALE 3/4" = 1"

Fig. 9

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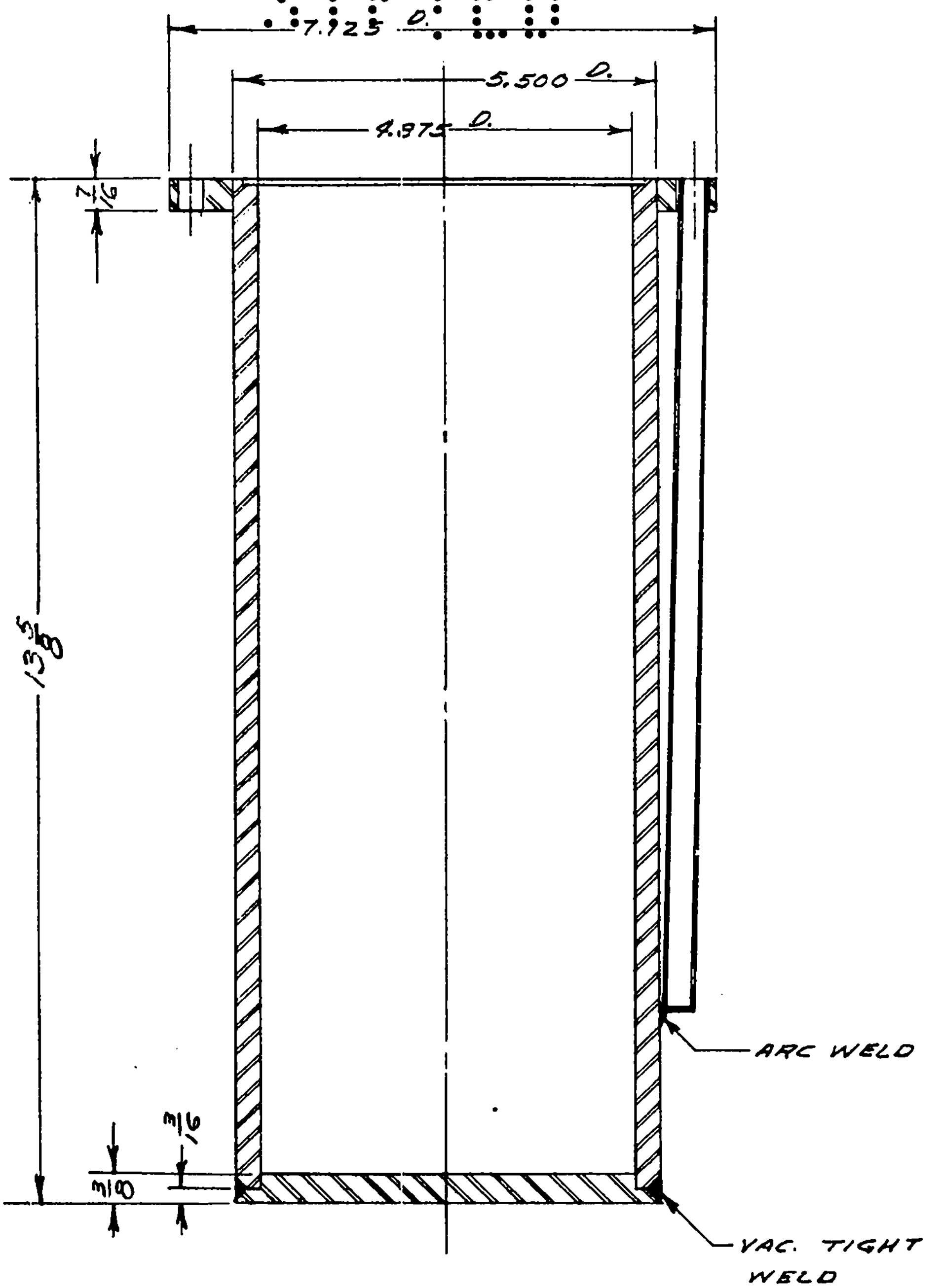

BOMB - 2 KILOGRAM

SCALE 1/2" = 1"

Fig. 10

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