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PRODUCTION OF CRUCIBLES FOR MELTING AND CASTING PLUTONIUM AND URANIUM-235

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ABSTRACT

The production of drain cast and solid cast magnesium oxide refractories is described in detail. Specifications for raw materials, procedures for preparation and casting of the slip, drying and burning of the cast refractories are given.

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1. <u>Introduction</u>

The slip casting process is adaptable to the fabrication of refractory shapes of such design, or with such thin sections, that they could not readily be made by the use of pressing or tamping techniques. The casting process also is quite useful and economical for the fabrication of special shapes when only a few pieces are required, as plaster molds can be made much quicker and cheaper than the steel dies used in the pressing process.

The prime incentive for the development of a method for slip casting magnesium oxide at Los Alamos was a request for crucibles 8 to 11 inches high, 3 to 5 inches in diameter, with a wall thickness of 1/16 to 1/8 inch.

Fabrication of magnesium oxide refractories by the slip casting process is not new, but as far as is known, its use in the past has been limited to the production of a few pieces of small size. Generally, the casting slips are prepared by very fine grinding of the magnesia in ethyl alcohol, and using relatively large amounts of hydrochloric acid to control the viscosity and casting rate of the slip. Such a process is not very suitable for quantity production of large shapes, owing to the rapid rate of evaporation of ethyl alcohol, the rapid deterioration of plaster molds due to attack by the hydrochloric acid, and the extremely high shrinkage of very finely ground magnesia.

In the process described in this report, water was used as the suspending medium, a very small amount of hydrochloric acid was used to control viscosity and casting rate, and some relatively coarse grain size magnesia was employed to reduce the shrinkage.

II. Raw Materials

A. Body Ingredients

Fused magnesium oxide grain purchased from the Norton Company, water, and hydrochloric acid were used in the slip preparation.

1. Chemical Specifications of the Magnesia Grain

Chemical specifications for the magnesium oxide were:

Material		Allowable Limits
SiO_2	-	between 1% and 1.5%
B	-	less than 200 ppm
Be	-	less than 5 ppm
Fe_2O_3	-	less than 0.25%
SiO_2/CaO	-	between 2/1 and 3/1

2. Grain Size Distribution of the Magnesia Grain

Three grain sizes of magnesia were used; i.e., minus 100 plus 200 mesh, minus 200 mesh, and minus 325 mesh. Grain size specifications of each size were as follows:

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a. <u>Minus 100 plus 200 Mesh Size</u>

Sieve Size, Mesh	Percent Retained
100	0 $^{+1}_{-0}$
200	92 ±8
thru 200	8 ±8

b. <u>Minus 200 Mesh Size</u>

Sieve Size, Mesh	Percent Retained
200	1 ±1
325	39 ±10
thru 325	60 ±10

c. <u>Minus 325 Mesh Size</u>

Sieve Size, Mesh	Percent Retained
325	0

Distilled water was used as the suspending medium, and a 1:1 water to acid solution of hydrochloric acid (37 to 38½ percent HCl) was used as the deflocculating agent to control viscosity and casting rate.

B. <u>Mold Ingredients</u>

U. S. Gypsum No. 1 pottery plaster and tap water were used in the preparation of the molds.

III. <u>Fabrication</u>

The steps involved in the fabrication of magnesia refractory

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shapes by the casting process are: 1) making the necessary molds; 2) milling of part of the magnesia grain; 3) slip preparation and casting, either drain or solid process; 4) drying; 5) burning.

A. Mold Fabrication

The plaster molds used were prepared by the usual methods using a water to plaster ratio of 75:100.

B. Milling

The minus 325 mesh grain was obtained as follows: 2000 grams of minus 200 mesh grain with 30 percent of water were charged into a one gallon porcelain pebble mill containing a charge of 4200 grams of porcelain balls ½ to 1 inch in diameter. The mill was run at a speed of 50 revolutions per minute for a period of 15 hours.

C. Slip Preparation and Casting

The composition and mixing technique used depended on whether the slip was to be used for drain or for solid casting. Exact compositions and mixing procedures are given below.

1. Drain Casting

In the drain casting process, the mold is filled with slip, and when the coating on the mold, formed by the removal of a small amount of water, reaches the desired thickness, the excess slip is poured out of the mold leaving the coating, which then shrinks slightly so that it can be readily removed.

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The most satisfactory slip composition for drain casting had the following composition:

Slip No.	Minus 325 Mesh MgO,%	Minus 100 Mesh, Plus 200 Mesh MgO,%	1:1 HCL (cc per 100 gms Dry Batch)	H_2O (% of Dry Batch Weight
32	60	40	0.0576	19.8

Procedure for preparation of the slip for drain casting was: Three percent additional distilled water was added to the mills after 15 hours milling time; the mills were then run for about 5 minutes to thoroughly mix the slurry. The slurry, which now had a water content of 33 percent of the dry weight of minus 325 mesh magnesia, was emptied from the mills and mixed for a few minutes by a variable speed mixer to remove the air bubbles. The slurry was covered with a damp cloth and allowed to stand for about 8 hours, when it was again mixed for a few minutes to break up the skin which had formed on the surface, after which it was again covered with a damp cloth and allowed to stand for about 16 hours. This made a total standing time of 24 hours after milling before the slurry was used to prepare the slip.

After the slurry had stood for the 24 hours, it was mixed by a variable speed mixer and the hydrochloric

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acid was added while mixing. The minus 100 mesh plus 200 mesh grain was then slowly added, and mixing was continued until all air bubbles had been removed from the slip. This process was used for the fabrication of "hollow ware" such as shown in Figures 1, 2, 3, 4, and 5.

2. Solid Casting

In the solid casting process, the molds are filled with slip and casting is allowed to continue until the cast piece completely fills the mold. Additional slip is added from time to time to keep the mold full as casting takes place. This process was used to fabricate stopper rods 3/4 inch in diameter and 14 inches long.

The composition of slip used for solid casting was:

Slip No.	Minus 325 Mesh MgO,%	Minus 100 Mesh, Plus 200 Mesh MgO,%	1:1 HCL (cc per 100 gms Dry Batch)	H_2O (% of Dry Batch Weight)
43	55	35	.0528	16.5

The mixing procedure for slip for solid casting was the same as that described for drain casting slip, except no additional water was added to the milled magnesia.

Slip prepared in either of the manners described above

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must be used within three or four hours after preparation. After this period of time, the slip gels and the casting characteristics are changed.

IV. Drying

Drain cast pieces were air dried at room temperature for at least 48 hours before burning. Solid cast pieces were air dried at room temperature overnight and then oven dried at a temperature of 80°C-90°C for at least 24 hours before burning.

V. Burning

Drain cast pieces were burned in an induction furnace having a graphite heating chamber whose inside dimensions were 20 inches in diameter and 28 inches high. The crucibles were set in an inverted position on unburned plates cast from the same mix as the crucibles. This minimized the warpage during the burning operation. Power for the furnace was supplied to a 32 inch diameter induction coil from a 100 KW, 3000 cycle motor generator set. A temperature of 1750°C was reached in 6½ to 7 hours with a maximum power input of 75 KW. After the peak temperature was reached, the power was cut off and the loaded furnace was allowed to cool naturally. The cooling period was 48 to 60 hours.

Pre-burning of solid cast pieces (stopper rods 3/4 inch in diameter, 14 inches long) to a temperature of 1200°C in 8 hours, using a resistance furnace, proved to be advisable. After pre-burning, the

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solid cast pieces were burned to 1750°C in induction furnaces in 1½ to 7 hours, depending on the furnace used. If the burning was done in the large furnace, the solid cast rods were bedded in a horizontal position in minus 28 plus 100 mesh magnesia grain on graphite shelves. The rods could be stacked three high on each shelf. When the burning was done in a small coil (8½ inch diameter setting chamber), they were placed on end in holes through a graphite block, the holes being the same length as the rods.

VI. Storage

After burning, the refractory pieces were wrapped in cellophane to minimize the pickup of dirt during storage.

VII. Results

At the present time, forty-nine magnesium oxide liners for the 1 kilogram reduction bomb, sixteen liners for the ½ kilogram bomb, and eight liners for the 2 kilogram bomb have been delivered. A majority of the liners have been used in reductions and results have been excellent.

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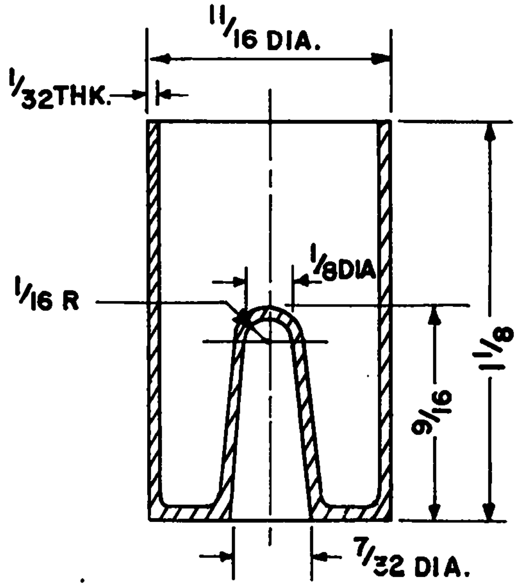

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Fig. 1 CRUCIBLE A-321

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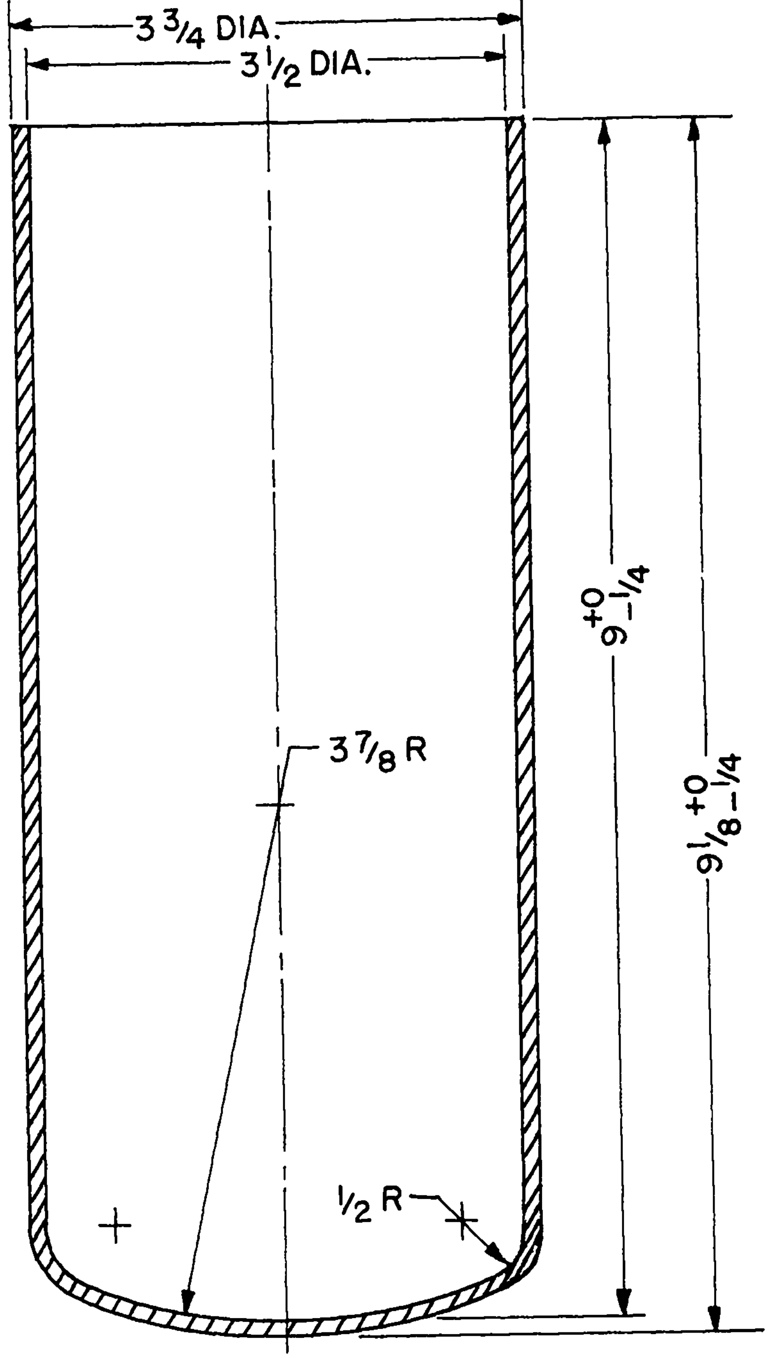

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Fig. 2. 1 KILO REDUCTION BOMB LINER NO. A-322-A

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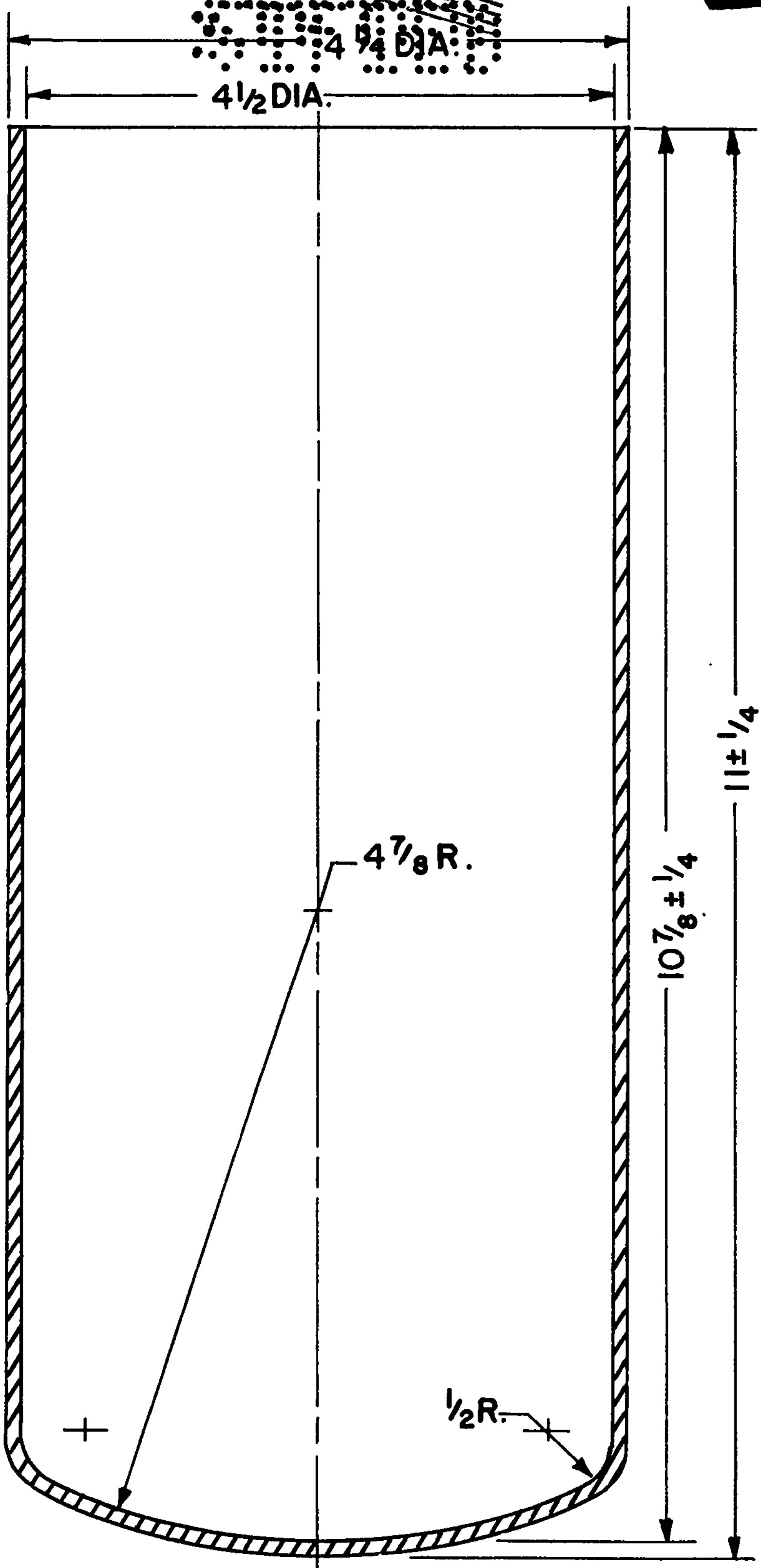

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Fig. 3 2 KILO REDUCTION BOMB LINER NO. A-332

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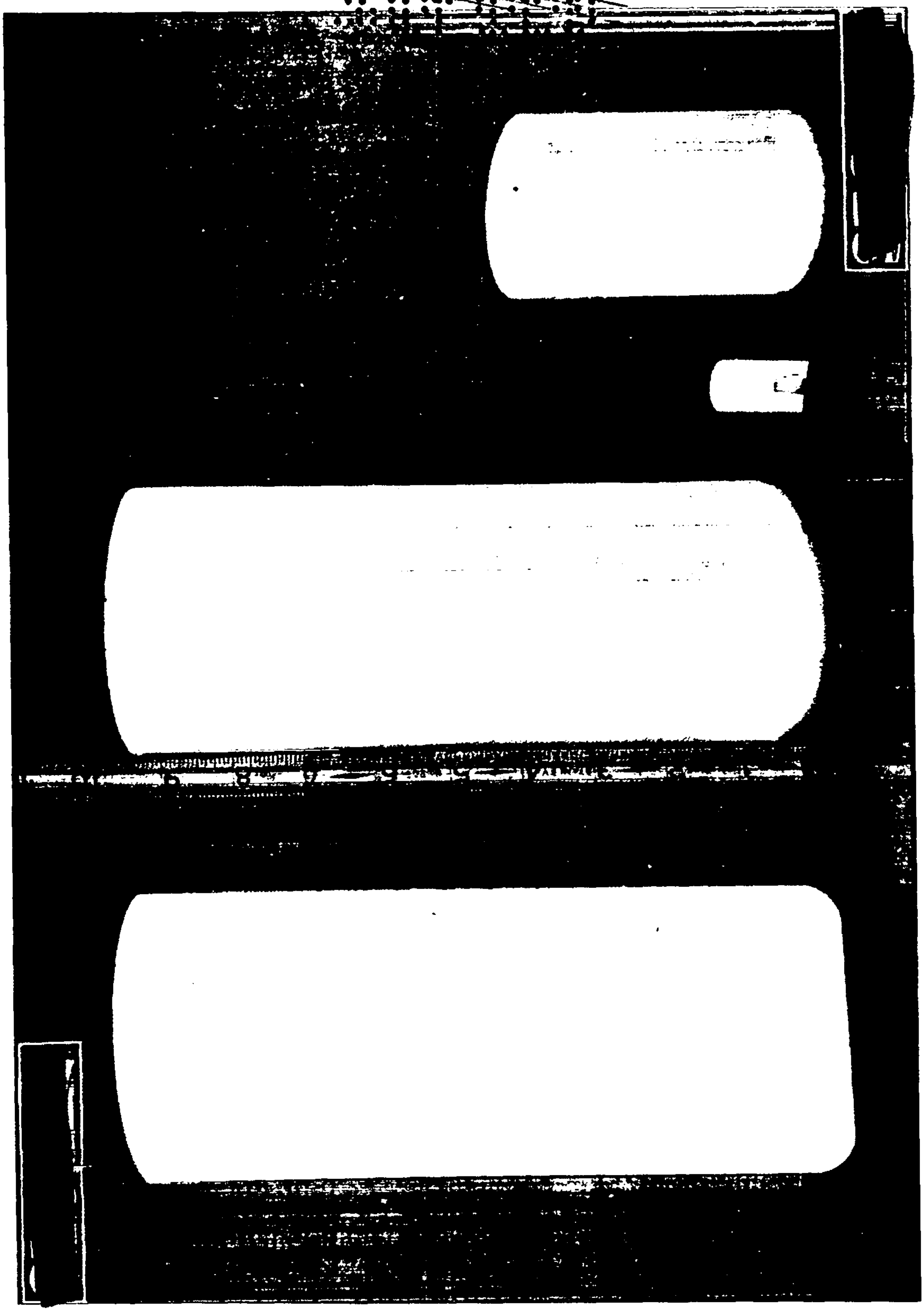

Fig. 4

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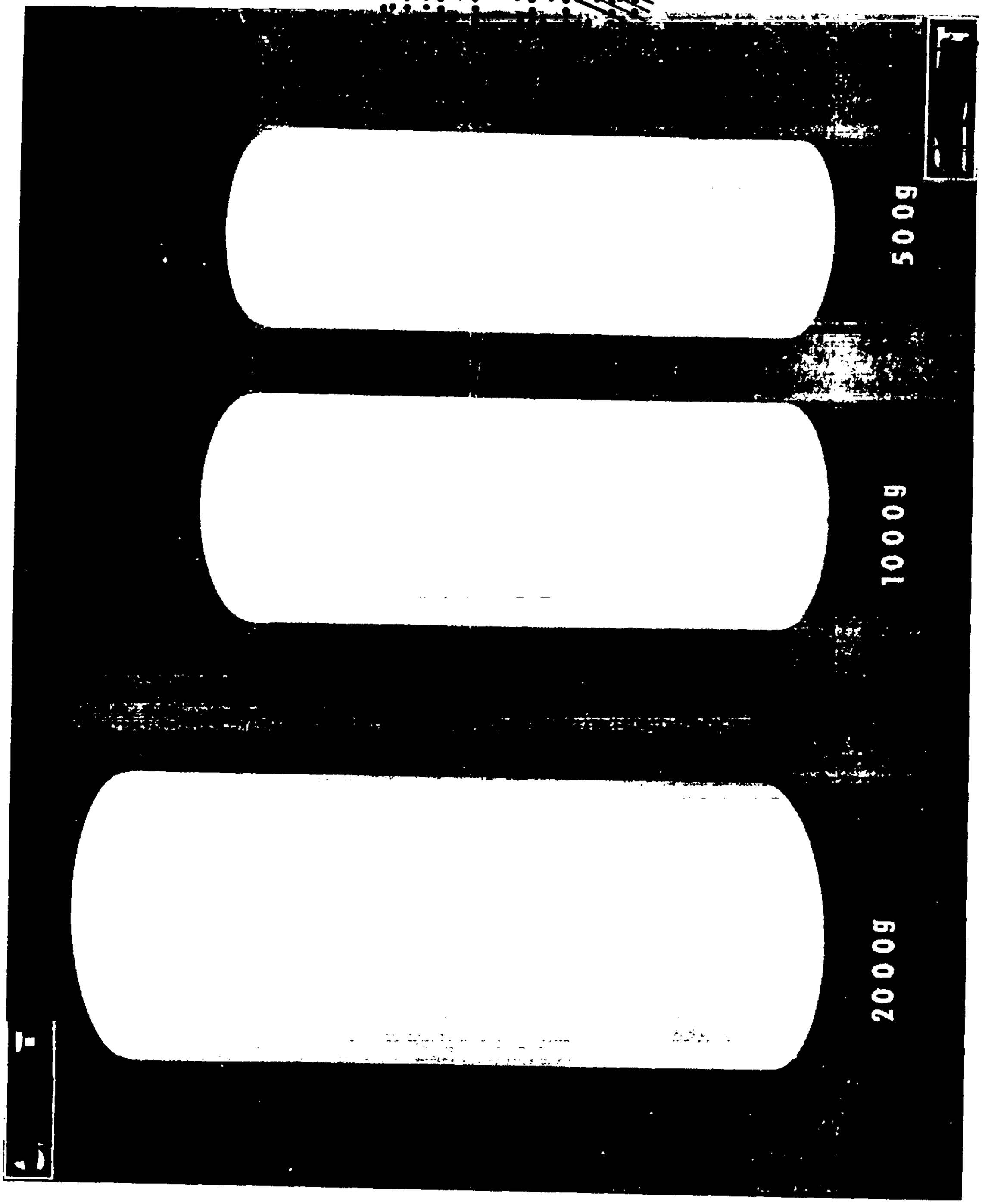

Fig. 5

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