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DEPARTMENT OF MINES
SOUTH AUSTRALIA

RESEARCH AND DEVELOPMENT BRANCH

CHEMICAL RESEARCH SECTION

D.M. 303/56

"RUBBER LINING INVESTIGATIONS - RADIUM HILL PROJECT .. PORT PIRIE"

SECOND REPORT

ISSUED BY:

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DATE:

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"REMOVAL OF FLOTATION OILS FROM RADIUM HILL URANIUM ORE CONCENTRATES BY FLASH HEATING"

by

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C O N T E N T S

S U M M A R Y

Flash heating has been shown to be no better than roasting the concentrates. Under conditions necessary to remove the greater percentage of the oil some loss in leaching efficiency was found. Heating the concentrates cannot be recommended as an economic method of oil removal. It is extremely unlikely that any other method can be found to remove the oil economically.

1. INTRODUCTION.

During an investigation of the removal of residual flotation oils from Radium Hill uranium ore concentrates, preliminary work by Madigan (1) showed that the only method which might have economic application was that of heating the concentrates sufficiently to burn the oil.

Roasting the concentrates in a muffle furnace at temperatures high enough to burn most of the oil, reduced the efficiency of subsequent leaching by approximately 4 percent.

It was suggested that flash heating might limit the effect to the surface of the concentrate particles and so minimise the deterioration in leaching efficiency.

This report covers the experimental work of flash heating Radium Hill ore concentrates.

2. MATERIAL EXAMINED.

2.1 - Radium Hill Uranium Ore Concentrate :

A representative sample received from Port Pirie Chemical Plant.

The sample was marked Composite SD

U_3O_8 = 0.84 percent
Moisture at 105°C = 9.7 percent

3. EQUIPMENT.

3.1 - Gas Burners :

Two straight burners, each containing two rows of 28 holes approximately one quarter of an inch apart, were placed in a horizontal plane parallel to each other and about 1.5 inches apart with the jets directed towards each other. This produced a sheet of flame 7 to 8 inches long between the burners. It was found necessary to use a mixture of compressed air and gas to obtain an oxidizing flame. The measured temperature of the flame was 800 to 850°C.

3.2 - Tubular Furnaces :

Two vertical furnaces were used during these tests (see Diagrams 1 & 2). The furnaces consisted of 2 inch diameter silica tubing and were heated by electrical resistance windings connected through a "Variac" to the mains supply. The furnaces differed mainly in size, the first being 15 inches long and the second 27.5 inches long. The temperature gradients of the furnaces were such that 5 inches of the First and11 inches of the second were at a temperature of 600°C or higher when the temperature measured by the thermocouple tip halfway down each furnace was 700°C.

3.3 - Sample Collector:

The apparatus shown in Diagram 3 was used to collect the treated sample without significant loss of fine material and at the same time allowed a current of air to be drawn down the furnace tube to prevent dry feed material being carried away from the top of the furnace by convection currents.

3.4 - Vibrating Feeder :

An electromagnetic vibrating feeder was used to introduce the dried and screened concentrates into the furnaces at a constant feed rate. A funnel shaped attachment, shown in Diagram 3, was screwed onto the feeder and extended a short distance into the mouth of the furnace tube to prevent loss of feed material.

4. EXPERIMENTAL PROCEDURE.

4.1 - Sample Preparation:

To obtain efficient removal of the oil it was desirable that individual particles should fall freely in an atmosphere containing oxygen. It was necessary therefore to dry and screen the concentrate.

The drying time was kept to a minimum and samples were screened as soon as sufficient moisture had been removed to prevent choking of the screen. Precautions were taken in this and all subsequent stages to prevent loss of fine material by dusting.

4.2 - Flash Heating :

4.2.1 .. Heating by Gas Burners -

This method of heating could not be readily evaluated in the laboratory because of the loss of the dry fine material due to the strong updraft caused by the burners. No quantitative measurements were made of oil removed by this method.

4.2.2... Heating in Tube Furnaces -

It was thought that an electrically heated furnace would eliminate one of the causes of the strong updraft found with the gas burners, namely, the introduction of large volumes of compressed air and gas. Preliminary tests, however, showed that sufficiently strong convection currents were produced in the furnace to cause serious loss of the finer material. The sample collection apparatus, shown in Diagram 3, was attached to the lower end of the furnace tube to collect the treated samples. Sufficient air to prevent the loss of fine material at the mouth of the furnace was drawn through the furnace by a vacuum pump. The second water trap was found to collect very little fine material if the feed rate was not excessive. The sample collector was used for all subsequent tests.

4.2.2.1 : Furnace No. 1 (15 inch Silica Tube)

Tests 1 to 6 inclusive were carried out in furnace number 1. Approximately 1000 g. of concentrate was dried in an oven and passed through a 30 mesh screen.

100 g. samples of this dried concentrate were used for all tests except number 6 in which 50 g. of the sample previously treated in test number 3 was used. Temperature measurements were made with the thermocouple tip halfway down the furnace tube. Samples were fed at approximately 12 g. a minute and allowed to fall down the furnace tube. The treated concentrate was collected in approximately 150 ml. water. After filtering and drying, the samples were extracted with ether and/or assayed for total carbon.

4.2.2.2 : Furnace No. 2 (27.5 inch Silica Tube)

Tests 7 to 11 were made in this furnace. Samples were prepared in a manner similar to those used in furnace number 1. 100 g. samples were used for all tests except number 11 in which a 500 g. sample was used. The method of treatment was similar to that described in 4.2.2.1.

4.3 - Leaching:

All leaches were carried out by the normal Radium Hill leaching procedure, i.e., using 750 lb. sulphuric acid per short ton, at a pulp density of 50 percent solids for 10 hours leaching time. The leached pulp was diluted with water and the liquor filtered. The residue was washed with water, dried at 105°C and weighed.

Leaching efficiencies were calculated from the uranium content of the dry residue.

5. RESULTS.

5.1 - Oil Removal and Leaching Efficiency :

Results obtained on the original concentrate and samples treated under various conditions are reported in Table I. Results obtained from the roasting of samples P_4 and P_5 in a muffle[1] are given for comparison with the flash heating tests.

TABLE I.

OIL REMOVAL AND LEACHING EFFICIENCY

Sample or Test	Method of Heating	Temperature °C.	Sample Wt. g.	Solvent Extraction		Carbon		Leaching Efficiency Per cent
				% extracted	% oil removed	% C.	% oil removed	
Original moist Sample	-	-	-	0.50	nil	0.91	nil	91
Original Sample	Dried for ½hr.	105	10	0.41	18	0.93	nil	n.d
"	Dried Overnight	105	1000	0.29	42	0.71	22	n.d.
P_4	Roasted for 3½ mins.	380 ± 20	100	0.015	97	0.43	53	87
P_5	"	350 ± 20	100	0.027	95	0.48	47	87
1	Furnace No. 1.	925 ± 10	100	0.08	84	n.d.	n.d.	n.d.
2	"	790 ± 10	100	0.10	80	n.d.	n.d.	n.d.
3	"	700 ± 10	100	0.10	80	0.42	54	87
4	"	600 ± 10	100	0.12	76	0.49	46	89
5	"	500 ± 10	100	0.15	70	n.d.	n.d.	n.d.
6	"	700 ± 15	50[a]	0.09	82	0.29	68	82
7	Furnace No. 2	900 ± 10	100	n.d.	n.d.	0.11	88	39
8	"	800 ± 10	100	n.d.	n.d.	0.13	86	n.d.
9	"	700 ± 10	100	n.d.	n.d.	0.20	78	n.d.
10	"	600 ± 10	100	n.d.	n.d.	0.42	54	82
11	"	700 ± 10	500	n.d.	n.d.	0.30	67	71

(a) 50 g. of treated sample from test 3.

n.d. not determined.

6. DISCUSSION.

It is clear that conditions which remove the greater percentage of the oil cause some loss in leaching efficiency. Flash heated samples appeared no better in this respect than roasted samples.

The roasted samples, because of the lack of oxygen and the lower ignition, temperature, appeared to char to some extent without complete burning of the carbon. Hence the high oil removal figures based on solvent extraction and lower figures calculated from the total carbon determination. For flash heated samples the two percentages were closer together indicating that more of the carbon was completely burnt. The best leaching efficiency obtained was 89 percent but the treated sample smelled of oil and would not wet completely with water indicating the presence of unremoved oil. A leaching efficiency not greater than 87 percent can be expected under conditions necessary to remove 80 to 90 percent of the oil.

7. CONCLUSIONS.

Flash heating of the concentrates appeared to have no advantage over roasting in a muffle. Most of the oil could be removed by heating but serious losses in leaching efficiency were found for the treated samples. A leaching efficiency of not more than 87 per cent can be expected under conditions necessary to remove 80 to 90 percent of the oil. Heat treatment of the concentrates, therefore, cannot be recommended as an economic method of removing oil from Radium Hill uranium ore concentrates. It is considered unlikely that any other economic method of removing oil could be found.

8. REFERENCES.

(1) Rubber Lining Investigations - Radium Hill Project, Port Pirie .. FIRST REPORT - March, 1956.

9. APPENDIX.

Analytical Methods

The Estimation of Flotation Oils in Ore Concentrates and Heat Treated Samples.

9.1 - Equipment :

9.1.1 .. Soxhlet Extractor -

A normal type soxhlet extractor was used to determine the amounts of ether soluble material in the original concentrate and in some of the heat treated samples.

9.1.2 .. Combustion Furnace -

Total carbon determinations were carried out in a standard organic combustion furnace.

9.2 - Analytical Procedure :

9.2.1 .. Solvent Extraction -

10 g. samples were extracted with redistilled ethyl ether in a soxhlet extractor. When the extraction was complete the ether was evaporated off and the residue weighed. This gave the percentage of extractable material in the sample calculated on a moisture free basis. The percentage of the oil removed was calculated from the extraction figure of 0.50 percent in the original concentrate as received.

9.2.2 .. Total Carbon Determination -

The carbon was burnt off in a combustion furnace at 800°C in a current of dry carbon dioxide free oxygen. The exit gases were dried and the carbon di oxide evolved was absorbed and weighed. From this the percentage of total carbon in the sample was found on a moisture free basis. The percentage of the oil removed was calculated from the total carbon figure of 0.91 percent in the original sample as received.

9.3 - Discussion:

Evaluation of the results was complicated by the fact that neither the solvent extraction nor total carbon figures showed with any certainty how much of the oil had been removed. Calculation of the percentage of the oil removed by using solvent extraction figures may lead to high results as it is probable that the solvent extractable oils burn first. When using the total carbon figure as a basis for the calculation low results are obtained as partially decomposed oils still contribute to the total carbon present. If it is assumed that the partially decomposed oils may not attack rubber then the true figures will lie somewhere between the two calculated percentages.

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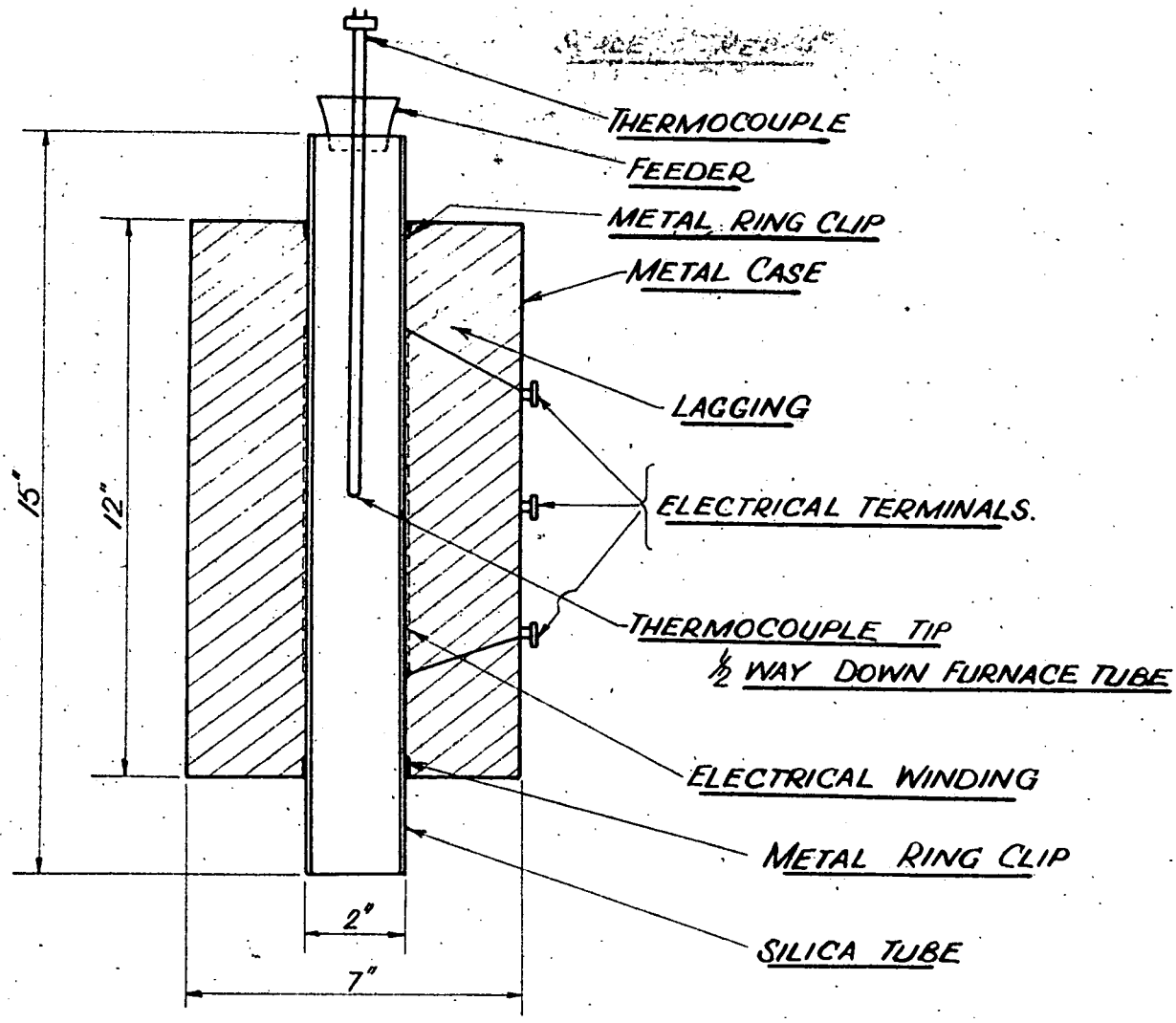

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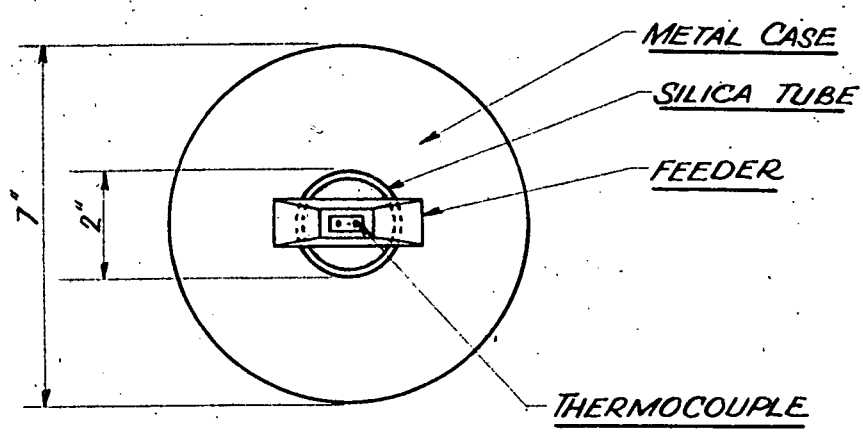

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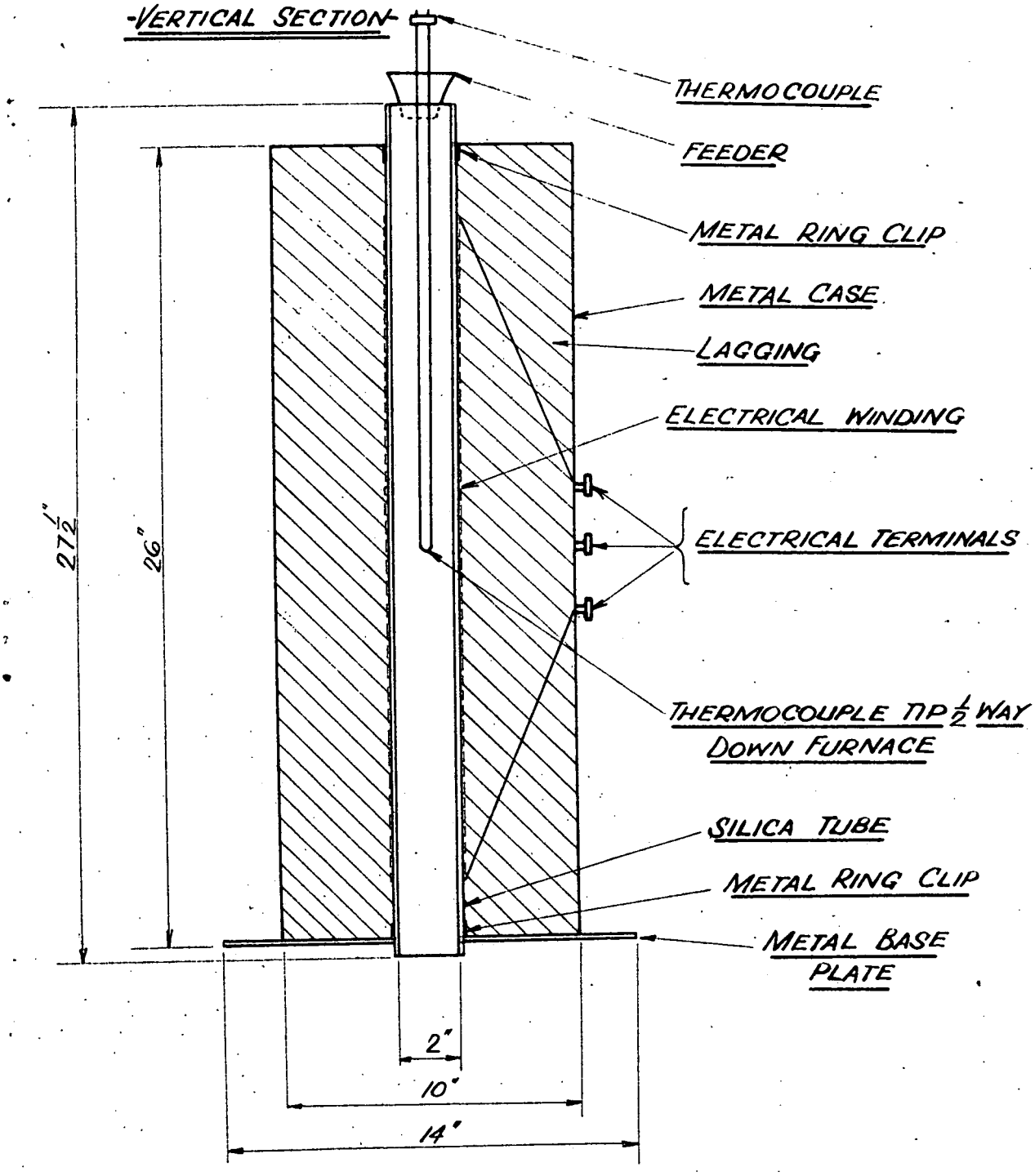

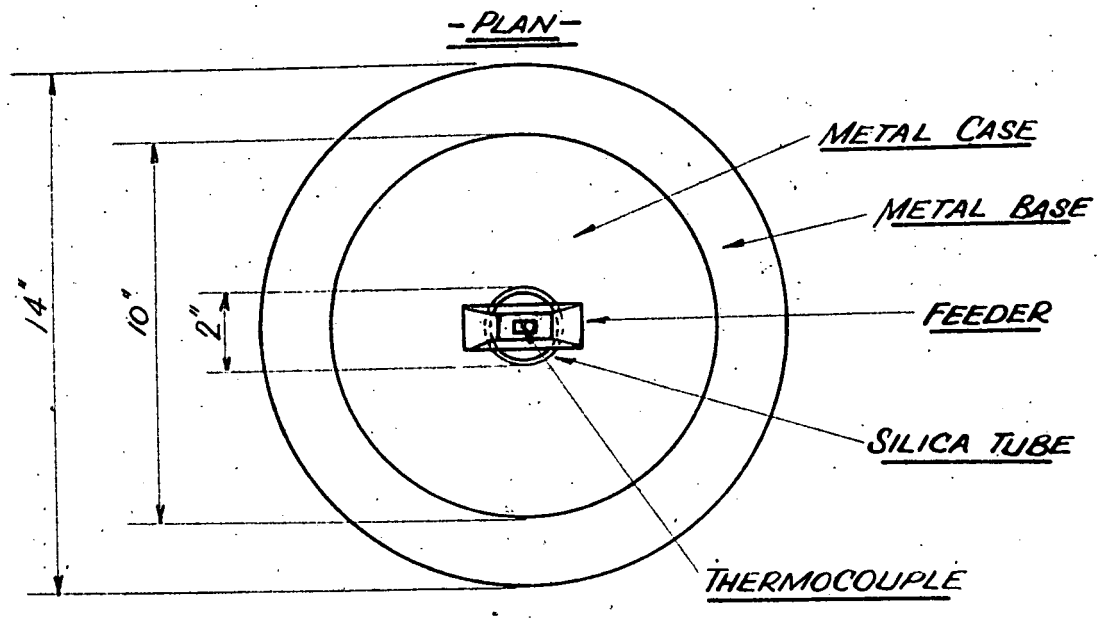

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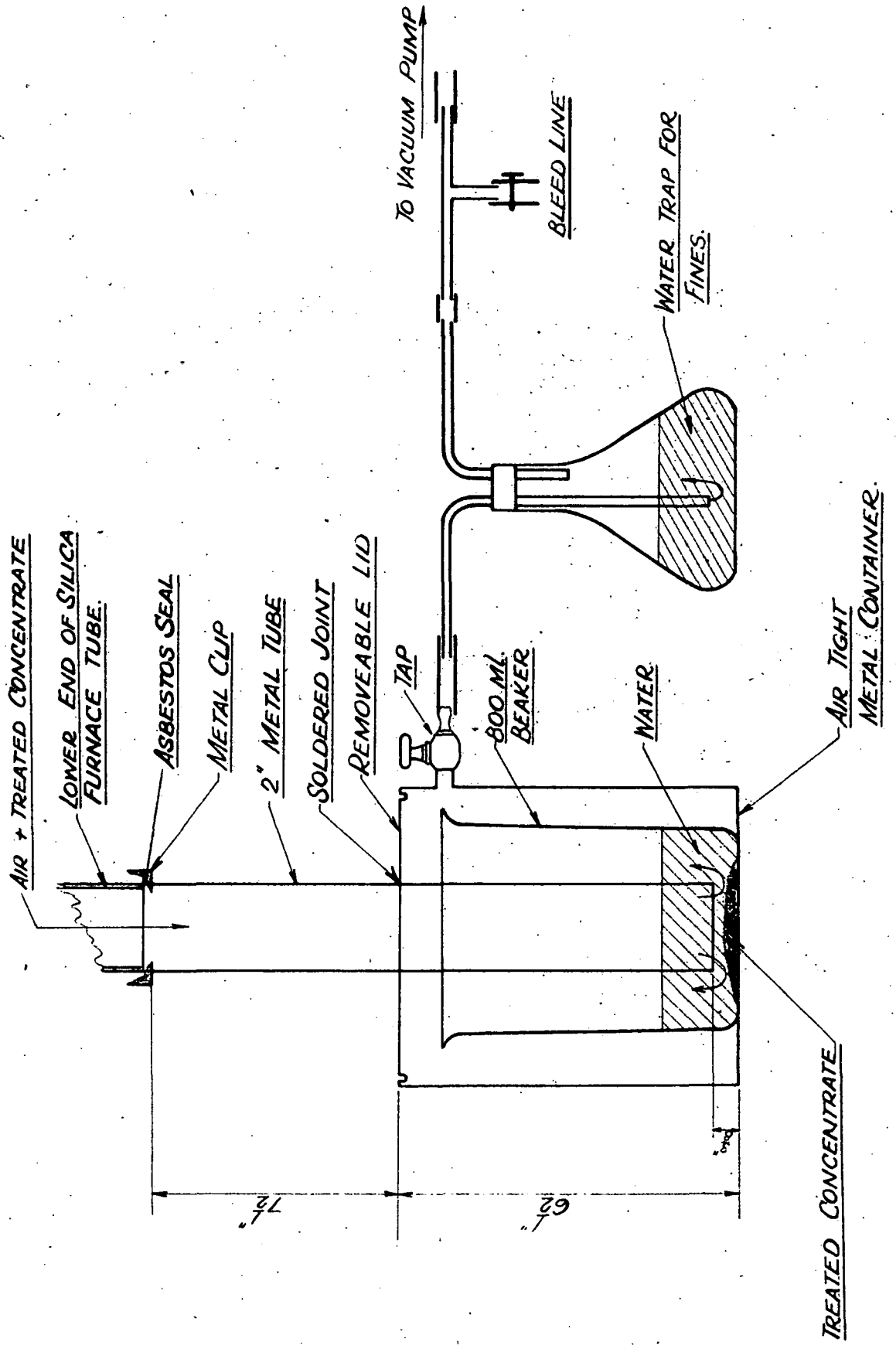

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