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NAVORD REPORT

6629

IMPROVED APPARATUS AND TECHNIQUE FOR THE MEASUREMENT OF THE  
VACUUM STABILITY OF EXPLOSIVES AT ELEVATED TEMPERATURES (U)

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NAVORD Report 6629

IMPROVED APPARATUS AND TECHNIQUE FOR THE MEASUREMENT  
OF THE VACUUM STABILITY OF EXPLOSIVES  
AT ELEVATED TEMPERATURES

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ABSTRACT: This report describes an improved constant temperature block which is used for studying the stability and compatibility of explosives at elevated temperatures. A very small area of the heating block is exposed while introducing or removing a sample from any one of the twelve sample positions. This is achieved by means of a rotating top.

A single piece all glass unit consisting of a sample tube and manometer is described for making vacuum stability measurements at elevated temperatures.

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U. S. NAVAL ORDNANCE LABORATORY  
White Oak, Silver Spring, Maryland

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12 March 1959

This report describes an improved constant temperature apparatus and technique for studying the stability of explosives at elevated temperatures. The apparatus and technique have proved useful in those projects where exposure to high temperatures would cause trouble with many conventional explosives and propellants. Other agencies working in these fields may find this report of definite value. Funds allocated under Task 301-644/-43006/08 were used for the work.

MELL A. FETERSON  
Captain, USN  
Commander

*Albert Lightbody*  
ALBERT LIGHTBODY  
By direction

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IMPROVED APPARATUS AND TECHNIQUE FOR THE MEASUREMENT  
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INTRODUCTION

In one of the programs of the Organic Chemistry Division there arose a need for studying the thermal decomposition of explosives at temperatures in the range of 200° to 300°C. Methods previously used (1) were found unsatisfactory because of the difficulty in maintaining a constant temperature in the existing apparatus and because ground glass joints could not be employed. This report describes an improved constant temperature apparatus, Figure 1, and the technique for making vacuum stability measurements at elevated temperatures.

PROCEDURE

The vacuum stability test at elevated temperatures is carried out in a sealed glass unit, Figure 2. These units are made in our Laboratory and each is calibrated before use. Generally one uses a 0.2 gram sample and a sample tube volume of about 30cc. After addition of the explosive to the sample tube, the open section is sealed just above the position where it joins the capillary tube. Before the glass sealing operation is carried out, the sample tube is placed inside a capped pipe section which is used as a protective device. A hole is made in the cap large enough for inserting the sample tube, Figure 3.

After the glass unit has been sealed the technique for making the stability measurement is similar to that previously described (2), (3).

HEATING BLOCK

The heating block, Figure 4, is made from a 7.8" diameter aluminum cylinder. Twelve equally spaced 1 1/8" diameter holes are machined in this cylinder 9" deep on a 6" diameter circle. In addition, three holes are drilled into the side of the block for a thermocouple, thermometer, and thermoregulator, Figure 8. This machined cylinder is heated electrically. Part 2 in Figure 5 is a circular slab of aluminum 14" in diameter and

1" thick. Twelve equally spaced holes are drilled to match those in part 1, and the two parts are bolted together. Part 2 has a recess on the top side for a three inch ball bearing. Leading out from each hole are slots upon which rests the horizontal part of the glass capillary, Figure 2. Part 3 shown in Figure 6 is made of the same material and dimensions as part 2. Part 3 is recessed at the center on the bottom side to receive the other half of the ball bearing. There is a 1 1/8" wide slot cut on a 6" diameter circle to allow for possible irregularities of the glass stability tubes. Two transite rings, Figures 6 and 7, are fitted over parts no. 2 and 3. The transite ring placed over part 2 contains slots to match those of part 2. Steel pins are used to line up the transite rings to parts 2 and 3 and to the outer container. Parts 1, 2, and 3 are mounted in an aluminum can made from 1/4" stock. This can is made in two parts. The inside diameter is several inches larger than parts 2 and 3 to provide ample space for insulation. Figure 8 shows the assembly of parts 1, 2 and 3.

Milled asbestos boards of various thickness are used for the insulation. Pieces were cut with 7.8" diameter holes in the middle to fit over part 1, while some solid disks were cut. All were made to fit snugly in the aluminum container. The apparatus was insulated so that all voids and air spaces were completely filled. Enough solid disks were used on the bottom and top to give adequate insulation. We used a 17" diameter container which allowed about 4 1/2" of insulation at the side of the heating block. The block rested on about 6" of insulation. After the top of the apparatus, consisting of part 3, insulation, and outside container was assembled, a segment was cut out in order to provide an opening for the tube and capillary. By aligning the top opening with a sample position, a tube and capillary could be placed in the heating block. An insulating plug (with a handle) was made for the top opening which was inserted when a stability determination was underway. This insulating plug is shown in Figure 9. Since parts 1 and 2 are bolted together and part 3 rests upon a ball bearing supported by part 2, the top of the block can be revolved 360°.

#### HEAT REGULATING CIRCUIT

The heating element consists of sixty feet of no. 16 Nichrome wire spirally wound around part 1 insulated with two layers of thin (1/64") asbestos paper. The spiral element is evenly spaced on part 1, Figure 4, covering most of the surface of the block.

An improved electronic heat regulating circuit, shown in Figure 10, has a choice of two constant voltages which eliminates the high heat surge and the wide temperature fluctuations experienced with the apparatus previously used (1). One circuit is on continuously and produces, by means of a step down transformer, a temperature just below that desired; the other circuit, controlled by a thermoregulator and a DPDT normally open relay provides an intermittent higher voltage which maintains the apparatus at the desired temperature.

### DISCUSSION

The constant temperature block described herein is a considerable improvement over the design previously used for measurements at 200°C or higher. During a six hour period, at 260°C (500°F) our improved apparatus maintained this temperature within  $\pm 0.7^\circ\text{C}$ . We have used the block successfully at 300°C and believe that it can be used effectively up to about 400°C, should the need arise.

The improved operating characteristics are achieved by better insulation, by a redesign which employs a rotating top, and by a new temperature regulating circuit. Only a very small area of the top, large enough for a single sample tube, is exposed when removing a sample tube or replacing one in the block. During this operation all of the other sample tubes are completely enclosed which is highly desirable from the safety standpoint. Also, temperature fluctuations are reduced to a minimum because of the small area uncovered. In the equipment previously used it is necessary to expose about one third of the area or more on the top when sample tubes are placed in or removed from the block.

After addition of the explosive to the sample tube, we have found it convenient to place the glass unit in a 100°C heating block for about two hours in order to dry the sample and the glassware before it is sealed. This operation is not necessary for an adequately dried sample and glass unit.

Each completed vacuum stability unit that has been evacuated and ready for test is allowed to stand at room temperature for a period of at least one hour before it is placed in the heating block. Any significant drop in the mercury level of the manometer will reveal a leak in the system.

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Readings of the mercury level in the manometer are taken as often as necessary to gain the desired information. In general, we neglect the first ten minute heating period as it does not reflect, in most instances, the thermal decomposition of the explosive under study.

ACKNOWLEDGMENT

We gratefully acknowledge the work of Joshua D. Upton. He not only contributed ideas toward the design, but also assembled the apparatus after the various parts were manufactured. The authors are indebted to Rolf Goderstad and his competent staff, who drew up plans for the fabrication of the apparatus and who also aided in many problems that arose.

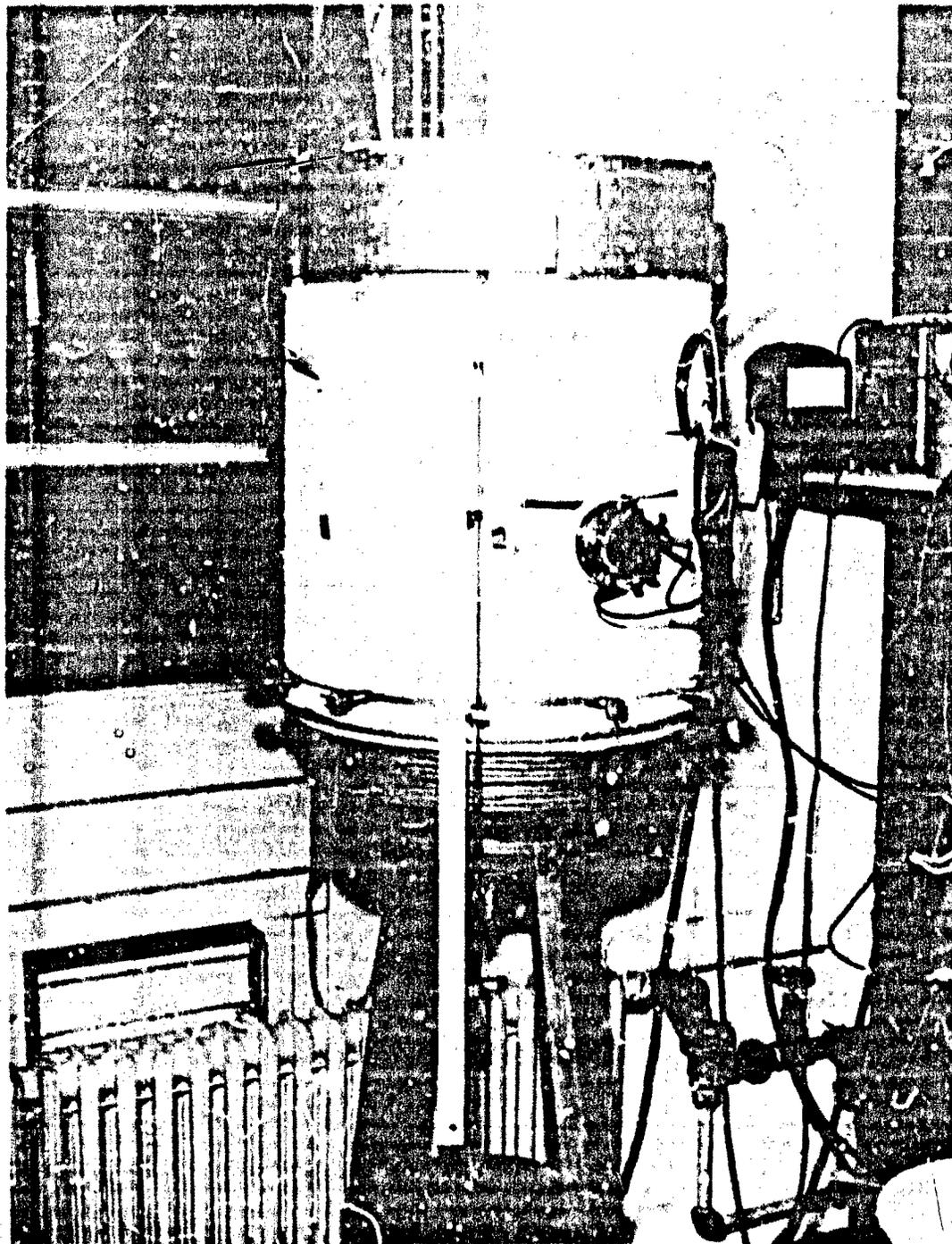


FIG. 1 CONSTANT TEMPERATURE BLOCK



FIG. 2 VACUUM STABILITY TUBE

NAVYON-15-1000-1000

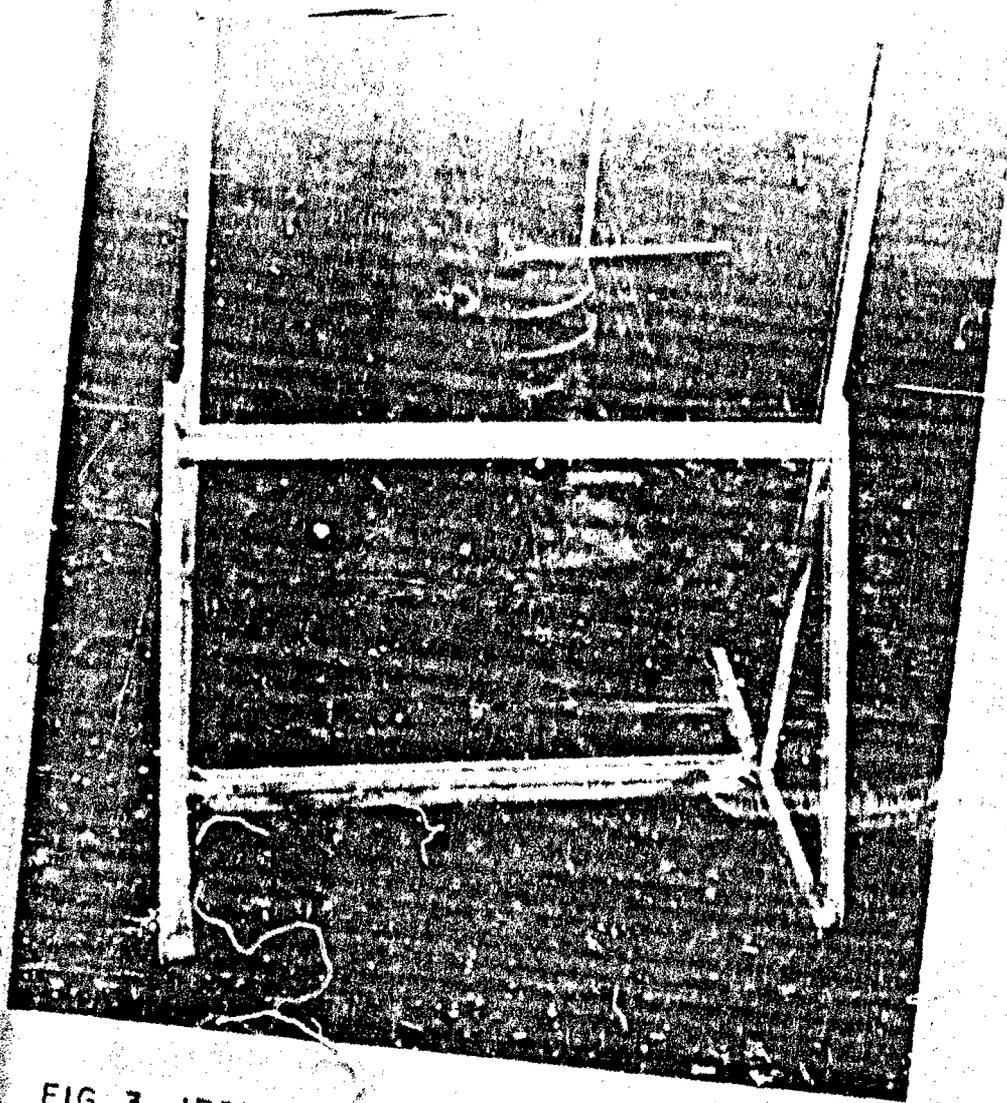


FIG. 3 IRON PIPE USED FOR SEALING TUBE

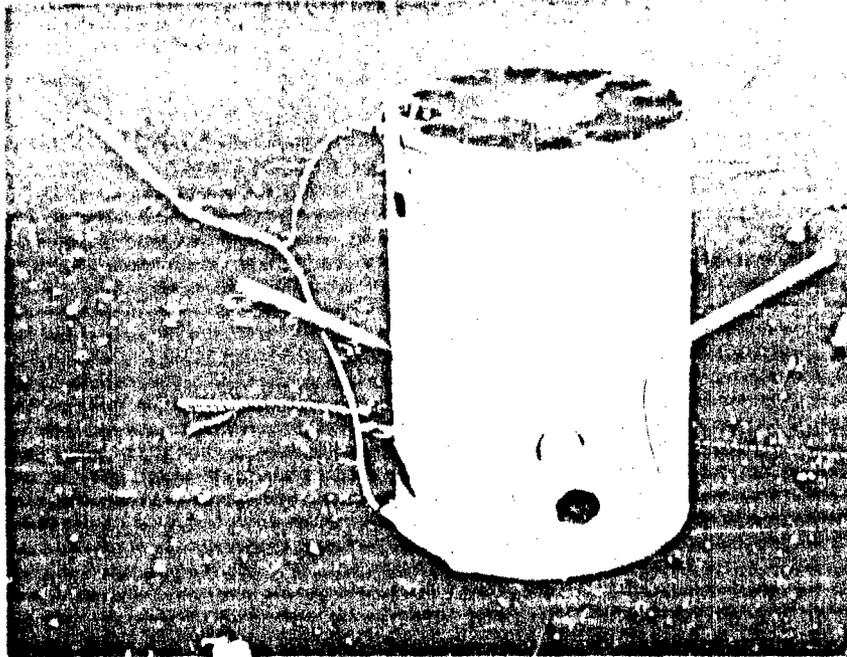


FIG. 4 HEATING BLOCK (PART 1)

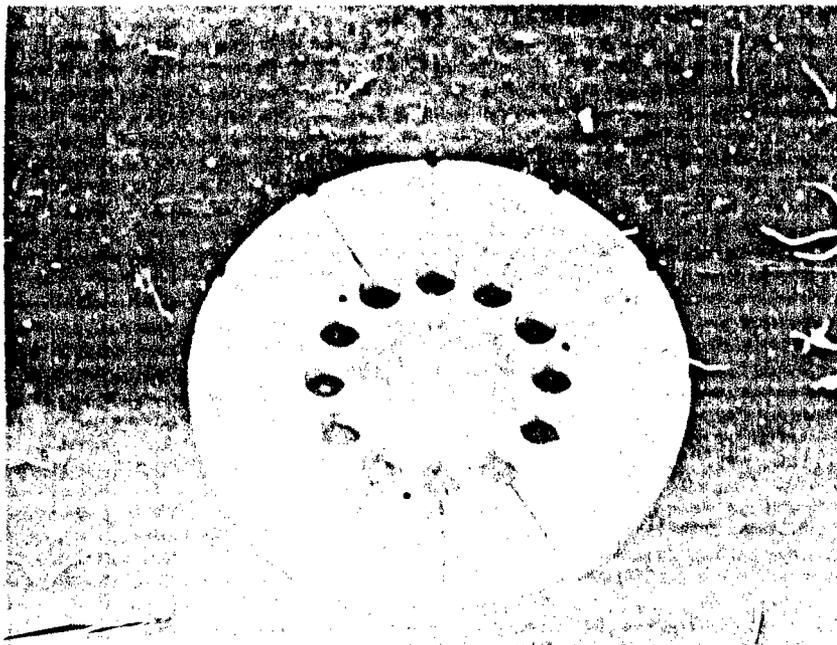


FIG. 5 HEATING BLOCK (PART 2)



FIG. 6 TOP OF APPARATUS REMOVED AND TURNED UPSIDE DOWN

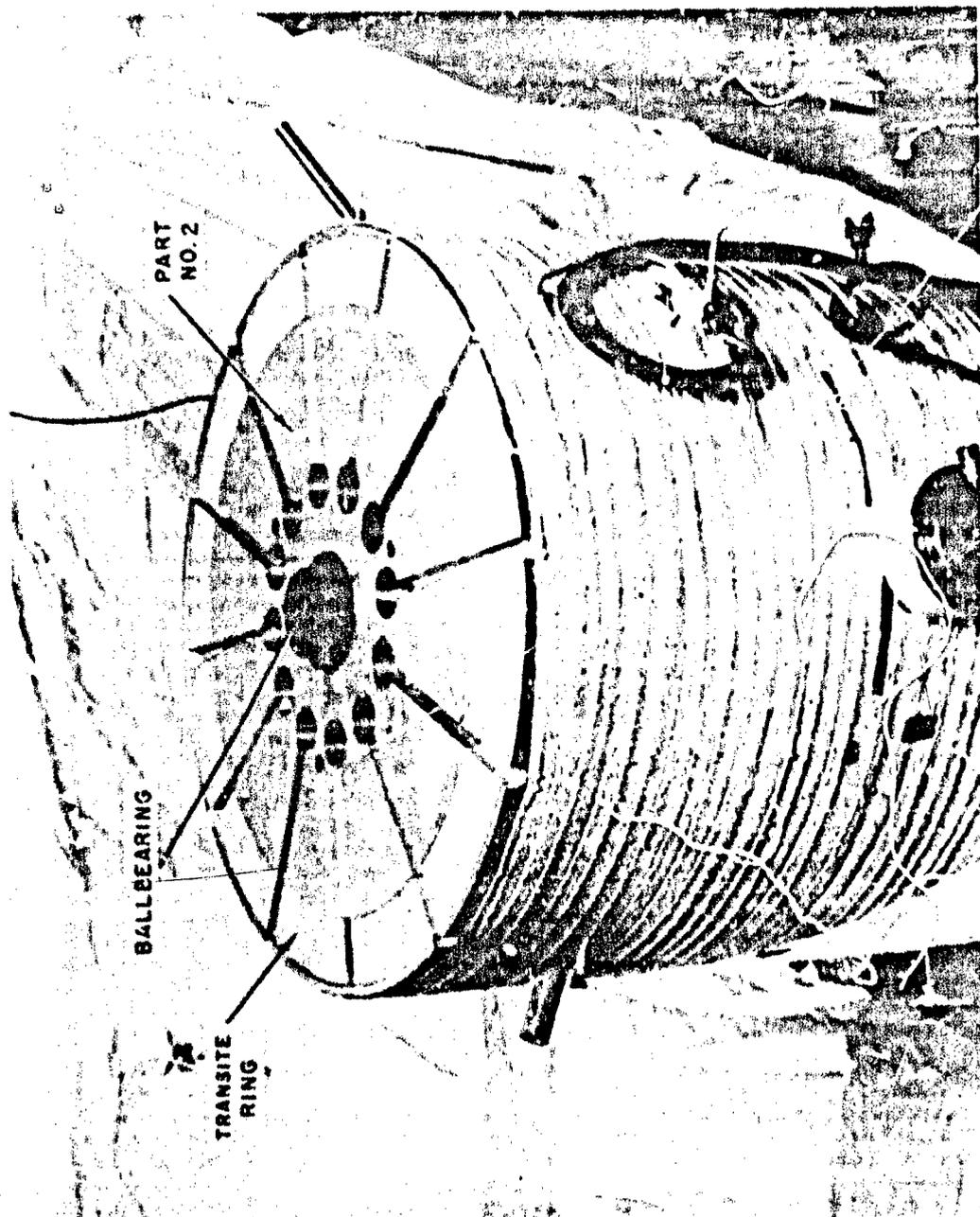


FIG. 7 TOP OF APPARATUS REMOVED

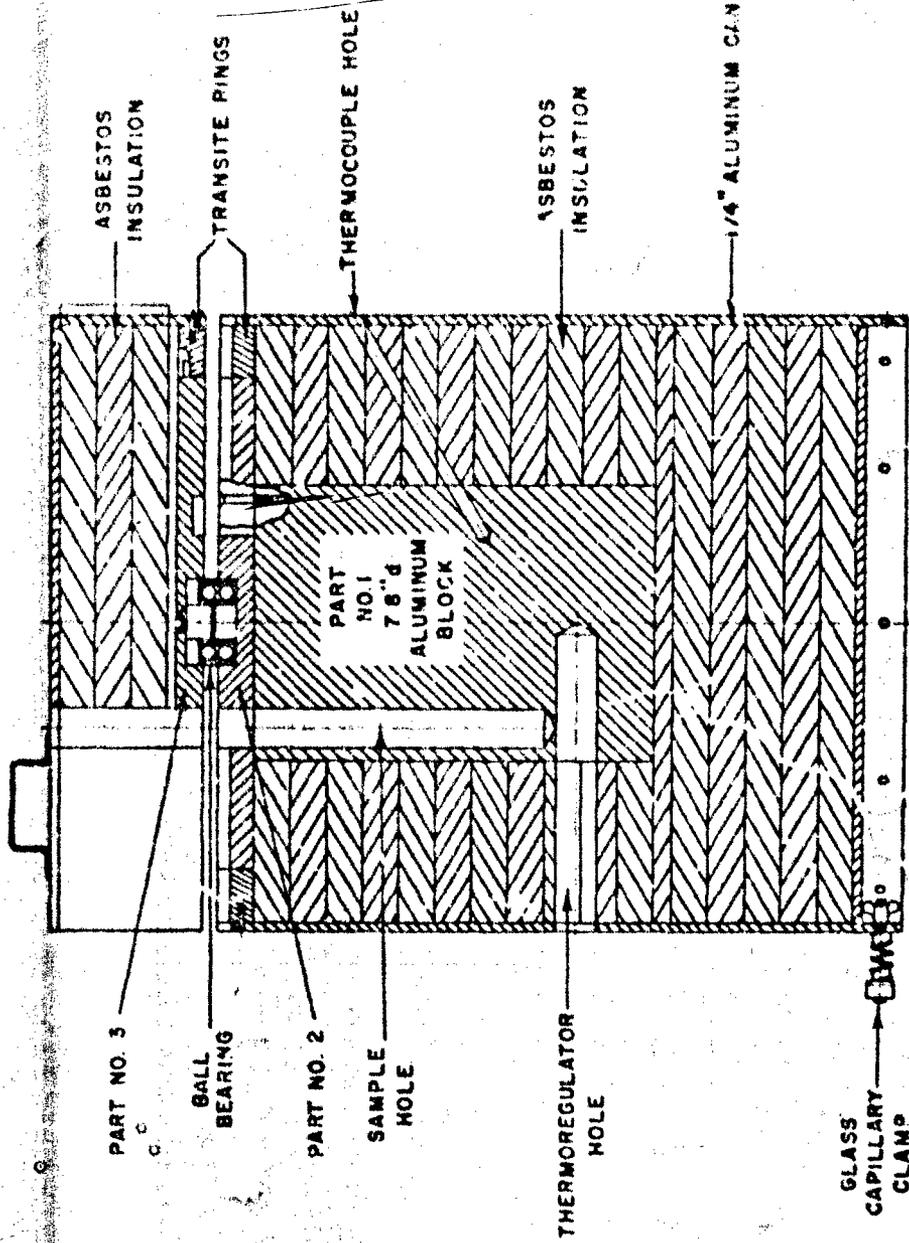


FIG. 8

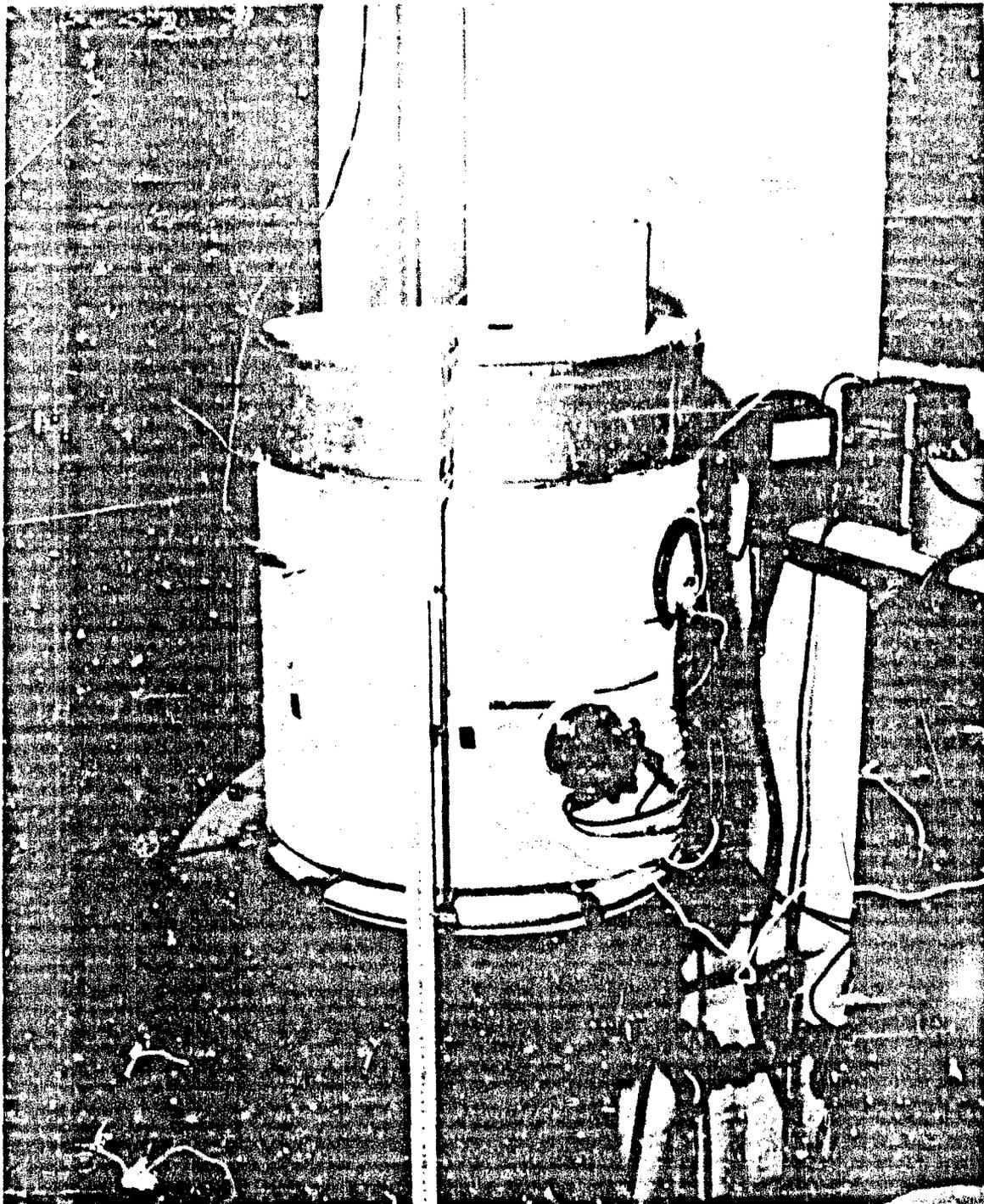


FIG. 9 STABILITY APPARATUS WITH PLUG REMOVED

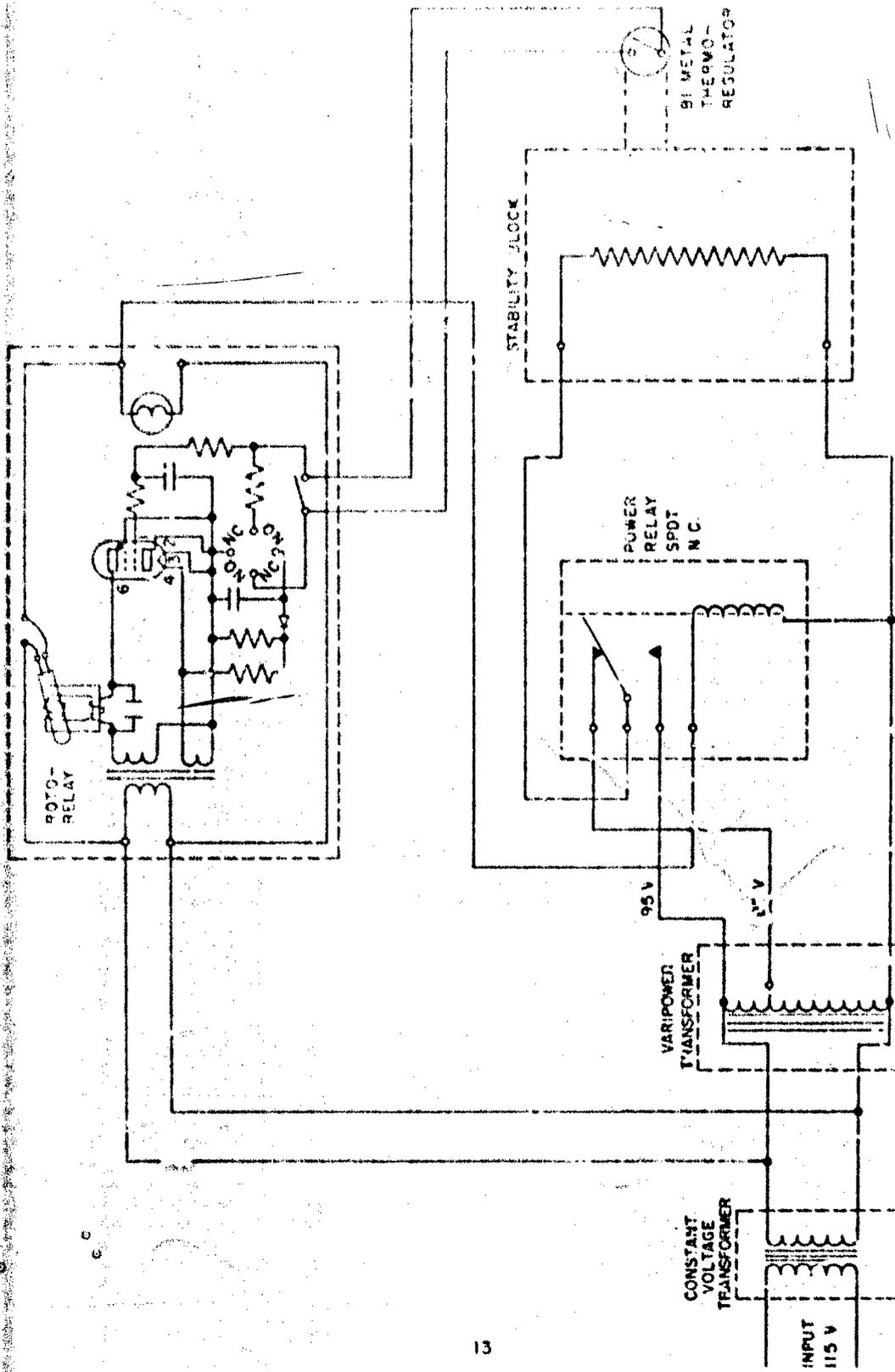


FIG. 10 HEAT REGULATING CONTROL CIRCUIT

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