NBS

Technical Note 141

CONTROLLED TEMPERATURE OIL BATHS FOR SATURATED STANDARD CELLS

PATRICK H. LOWRIE, JR.

U. S. DEPARTMENT OF COMMERCE
NATIONAL BUREAU OF STANDARDS

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AUGUST 1962

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ABSTRACT

Two oil baths for the temperature control of saturated standard cells have been designed and fabricated at the Boulder Laboratories of the National Bureau of Standards for operation at 28°C and 35°C respectively. Short term control to better than ± 0.001°C with day-to-day variations no greater than 0.002°C has been achieved with the use of a mercury-toluene thermoregulator incorporating a temperature anticipating device. The circulating system limits temperature gradients in the oil to less than 0.001°C across any 10 inch section. The baths incorporate pre-heat and drain tanks as well as the main temperature regulated tank to facilitate the insertion and removal of cells and to minimize oil spillage.

1. Introduction

For over fifty years the basic voltage standard has been the saturated Weston standard cell. This cell consists of a cadmium amalgam electrode and a mercury-mercurous sulfate electrode in a saturated solution of cadmium sulfate and is housed in a sealed glass H-tube [1] to [8]. Saturated cells are used as voltage standards because the average voltage of a group of these cells has been found to remain constant to within a few microvolts over many decades.

The cells present a problem, however, in that they are very sensitive to temperature, movement, and light. At a temperature of 28 °C the emf of a saturated cell will change approximately 50 microvolts per degree C, and if there is a temperature difference between the two legs of the cell, the emf may vary by as much as 300 microvolts per degree C of temperature difference. If a cell experiences vibration, the emf will vary provided the vibration is strong enough to disturb the free mercury or amalgam. If a cell is tipped more than about 45° it very likely will become unstable and therefore useless as a standard.[1] If a cell is exposed to daylight or fluorescent light for an extended period it may be permanently damaged. Exposure to direct sunlight should definitely be avoided. There is some evidence that incandescent light is less injurious to cells than is fluorescent light or daylight [8], but as no quantitative information of the effect of light on cells is available at present, the cells are generally protected from all sources of light.

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[1] Recently cells have been marketed which are claimed to be unaffected by tipping or jarring.

Though many baths performing the functions described below have been fabricated, apparently very little has been published regarding them since the very early papers [1] [2]. This paper describes two baths that were designed and built at the Boulder Laboratories for use at 28°C and 35°C respectively. These embody many of the features of similar baths already in use in the Standard Cell Section, NBS, Washington. The problems encountered and the techniques utilized in the design, fabrication, and adjustment of the Boulder baths are discussed.

2. Requirements

From the discussion above, it can be seen that there are three primary requirements of any enclosure for saturated standard cells. These are (1) close temperature control with temperature gradients reduced to a minimum (to minimize temperature differentials between the legs at any contained cell), (2) stability of the medium (for oil this means minimum turbulence consistent with small gradients), and (3) protection from light of the space containing the cells. In addition to these requirements, the baths should have excellent reliability, adequate capacity, and should be designed for maximum convenience. The specific requirements of the Boulder Laboratories were as follows: The baths should be capable of containing up to twelve groups of cells each, with up to six cells per group. The cyclic variations in temperature should not exceed 0.002°C. However, the temperature variation from day to day may be as great as 0.005°C provided the average temperature does not change from week to week. Provision should be made for making voltage measurements of the cells and for making temperature measurements of the oil with minimum disturbance to the cells.

Since none of the commercially available baths answered all of the needs of the National Bureau of Standards, Boulder Laboratories, it was decided that the baths should be fabricated at the laboratories. After a study had been made of the design of the oil baths in use at NBS, Washington, the decision was made to design a somewhat different bath. In addition to the features included in the Washington baths, the Boulder baths include a pre-heating tank[2] and drain tank.

3. Description

3.1 General

Each bath (figure 1) was designed as a complete unit incorporating the main oil tank, pre-heating tank, drain tank, and all control and circulating equipment. The outer wall of the bath and most other parts are fabricated of sheet stainless steel. The bath rests on a supporting framework of angle iron. The lid (figure 2) consists of two black, hard anodized aluminum sheets separated by one inch of polystyrene foam (figure 3). It is designed so that only a small section need be opened when measurements are being made. However, one entire side of the top of the bath may be exposed when cells are being transferred to and from the oil. The lid is also easily removable so that the bath may be completely exposed for repair should the need arise.

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[2] The author is indebted to David Ramaley and John F. Shafer for their suggestion that a pre-heater be incorporated in the design.

FIGURE 1. THE STANDARD CELL OIL BATHS AT NBS BOULDER

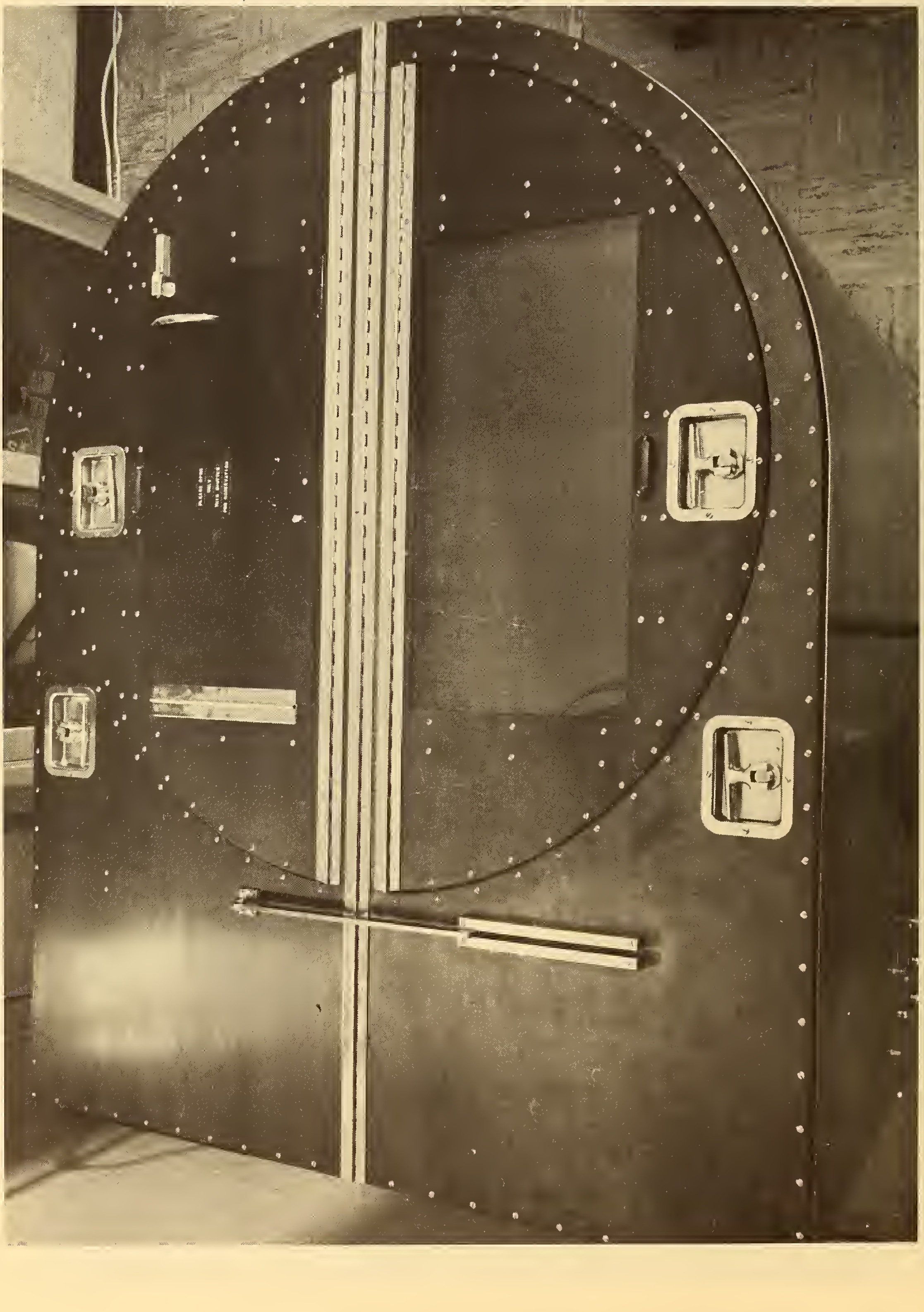

FIGURE 2. TOP VIEW OF 28°C OIL BATH

FIGURE 3. 35°C BATH COVER WITH ONE SIDE REMOVED TO SHOW POLYSTYRENE FOAM FILLER

3.2 Main Tank, Baffle, and Rack

The main tank is thirty-six inches in diameter, twenty-five inches high, and holds approximately ninety-five gallons of clear mineral oil. It is insulated on the bottom and sides by two or more inches of crushed cork and on top by the air space between the oil and the lid as well as by the insulation within the lid. Within the tank is a cylindrical baffle, as shown in figure 4, twenty-eight inches in diameter, open at both top and bottom. The baffle serves to control the flow of the oil in such a way as to minimize temperature gradients. In operation the oil is drawn down through the center of the bath, passes under the baffle walls, and circulates upward outside the baffle. The area between the baffle and the wall of the tank is less than that contained within the baffle; as a result, the oil moves faster with higher turbulence as it moves up outside the baffle. The speed of the oil is then reduced as it moves down over the cells, and the turbulence is decreased. Thus both thorough mixing and low effective turbulence are achieved. Bolted to the top of the baffle is the rack which holds the baskets containing the cell groups (figure 5). The baskets are hung on the rack in such a way as to place the center of gravity of the group of cells well below the points of suspension. This reduces the possibility of accidentally tipping the group. The baskets are provided with handles so that the groups may be easily handled during immersion in or removal from the oil (figure 6).

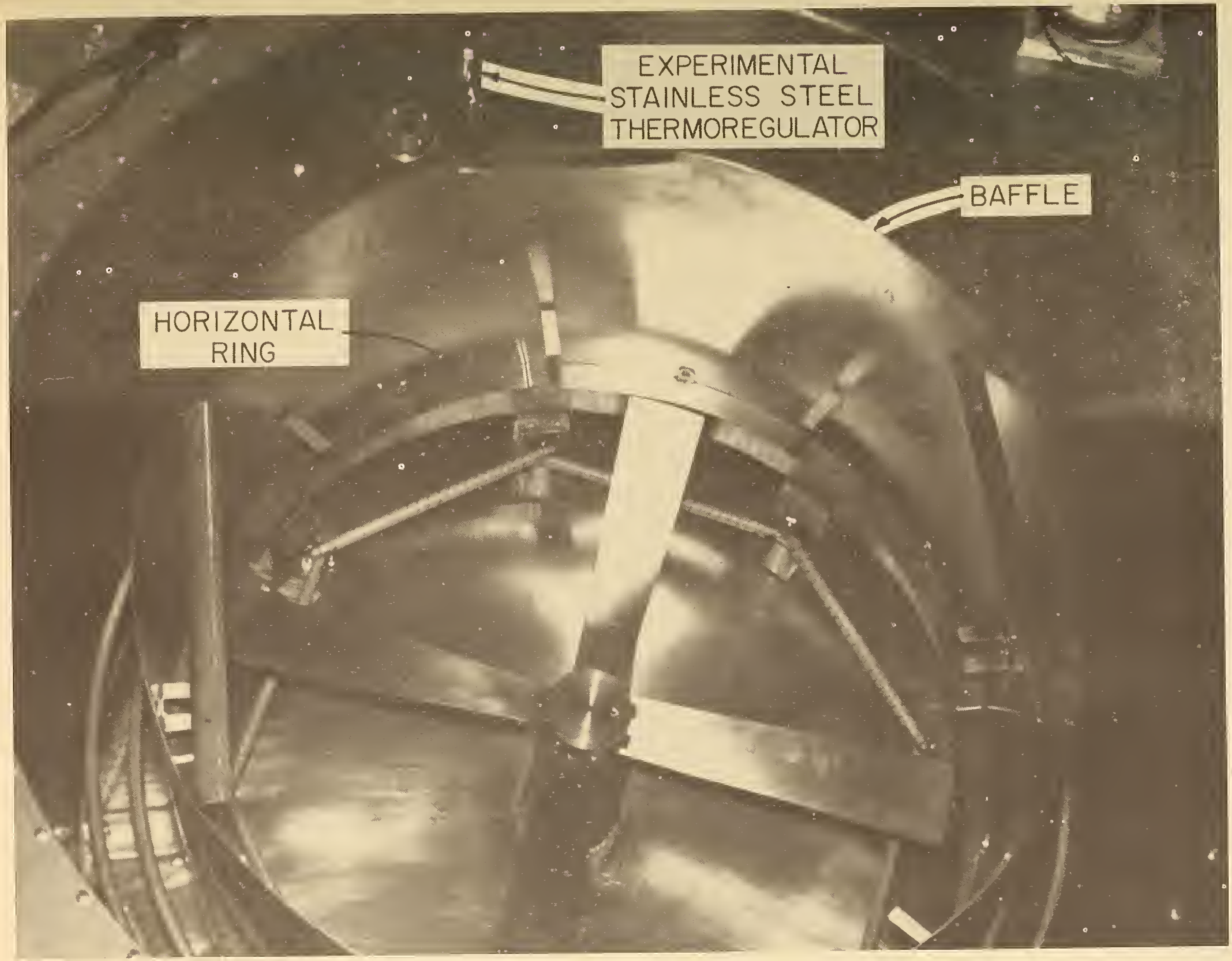

FIGURE 4. CLOSE-UP OF 35°C BATH DURING CONSTRUCTION

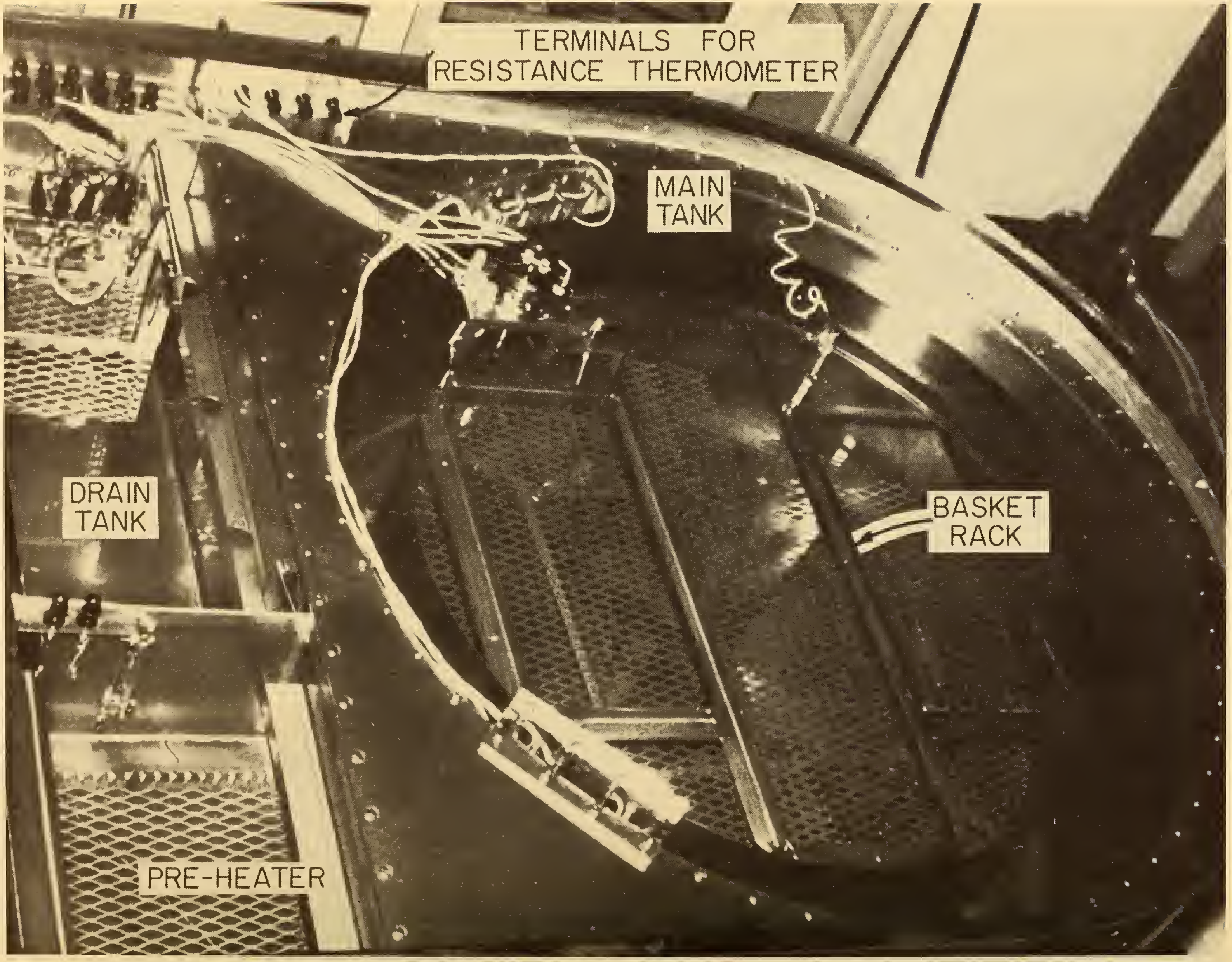

FIGURE 5. 28°C BATH WITH COVER REMOVED

FIGURE 6. STANDARD CELL BASKET

3.3 The Heater

The heater element is made up of approximately 50 ohms of number 22 Nichrome wire. It is wound between two hoops of insulating material to form an open grid through which the oil passes (figure 7). This type of construction minimizes the heat capacity of the heater, allowing it to operate at very nearly the temperature of the oil and to provide the most efficient heat transfer to the oil. The heater is placed at the bottom of the baffle so that the oil after passing through the heater has the maximum possible distance in which to mix before passing over the cells. The heater has current in it continuously; the action of the thermoregulator serves only to control the amount of current.

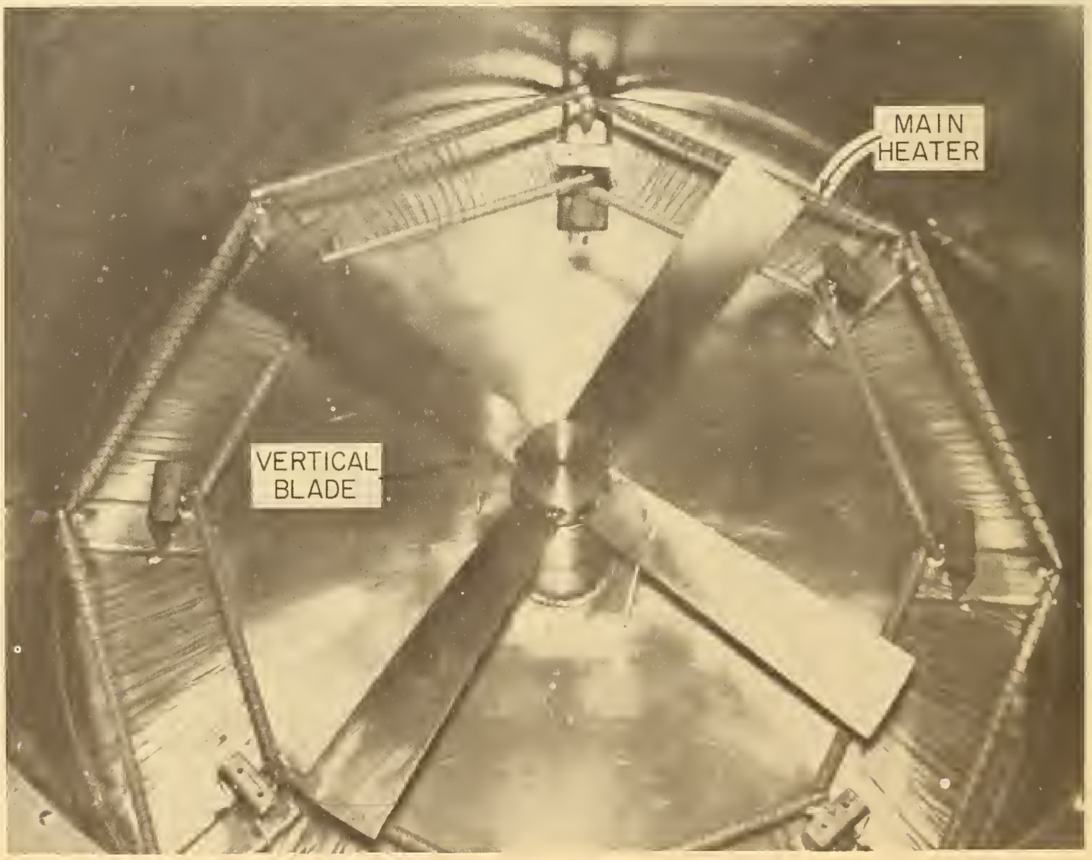

FIGURE 7. CLOSE-UP OF 35°C BATH DURING CONSTRUCTION

3.4 The Circulating System

During the initial experimentation with stirring systems, it was found that small, high speed impellors were unsatisfactory in that they introduced considerable heat into the oil. Since one of the baths was to operate at an oil temperature only five degrees Celsius above ambient, uncontrolled sources of heat could not be tolerated. At the suggestion of Dr. Walter J. Hamer, a larger low speed stirrer was designed. Hence, the oil is circulated by a four-blade stirrer, 25 inches in diameter, rotating at 55 rpm. A four-blade propeller was used as a stirrer because of its efficiency advantage over the two-blade type [9]. The shaft extends through the bottom of the tank to a 20 to 1 gear reductor which is connected to a 1/4 hp motor by means of a belt. Oil leakage through the shaft bearing is prevented by means of a mercury seal (figure 8). The motor is shock mounted on a platform under the control circuits section of the bath. It was originally connected to the gear reductor by a belt to facilitate changing propeller speed during the initial experimentation. Since the belt also serves to help isolate any motor vibration from the cells, it was retained in the final design. The motor is of the induction type and has sealed ball bearings. A fan is affixed to the motor shaft to assist in cooling both the motor and the enclosed area under the bath. The platform on which the motor rests was designed to be removable, so that the motor could be mounted on the floor or elsewhere in the event that the vibration was too great in its present position. This, however, was found to be unnecessary.

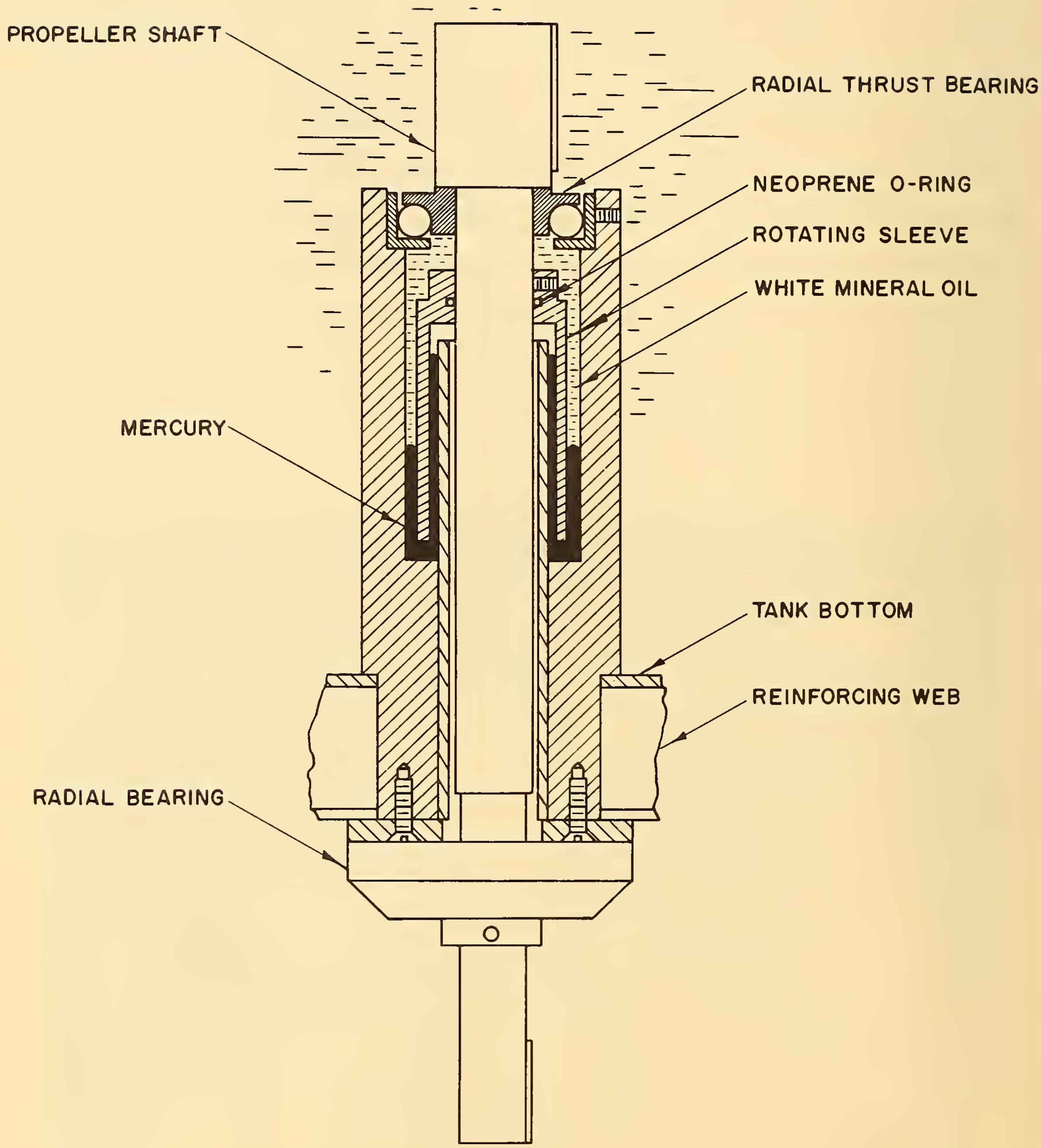

FIGURE 8. PROPELLER SHAFT AND MERCURY SEAL ASSEMBLY

Originally the inside of the baffle wall was smooth, and a gap of approximately one and one-half inches existed between the wall and the tip of the propeller blade. It was found that this configuration allowed the oil to slip past the tips of the blades and thus to recirculate without passing through the heater. This caused a rather large temperature gradient. The problem was solved by affixing a horizontal ring to the inside of the baffle at the level of the propeller (figure 4). This ring projects about one inch from the baffle wall and effectively prevents the oil from circulating up between the tips of the blades and the baffle wall. The efficiency of the propeller was further increased by the addition of two vertical blades, welded to the underside of opposing horizontal blades, as shown in figure 7, to thrust the oil toward the wall of the tank after it is drawn down through the horizontal blades.

The temperature gradients were determined by establishing the minimum distance between two points in the oil which, when probed with a platinum resistance thermometer, exhibited a temperature difference of 0.001°C. After completing the modifications described above, this distance was found to exceed 10 inches throughout both baths.

3.5 Thermoregulator

The temperature of the oil is controlled by a thermoregulator of the mercury-toluene type similar to that described in [1]. In this type of thermostat the mercury serves the two functions of sealing the toluene in the regulator and of providing a conducting path between the electrical contacts. Any expansion of the mercury due to a change in temperature is so small compared to that occurring in the toluene that it may be disregarded provided the bulk of the mercury column is under the oil. The amount of mercury exposed to the air should of course be minimized, since changes in air temperature could cause erratic action of the regulator. Toluene was chosen as the expansion element for a number of reasons. It has a high temperature coefficient of expansion (over six times that of mercury)[10], relatively large surface tension (though quite small as compared to that of mercury), and acceptably low volatility. The regulator was first constructed of stainless steel tubing (figure 4) with the hope that the advantages of greater safety and convenience of handling and the higher heat conduction of stainless steel, as compared to pyrex, would not be off-set by the considerably lower dimensional stability of the steel. It consisted of 1/2 inch o. d. tubing bent in the form of a double, concentric hoop which was closed at one end by a plug and at the other by a rather elaborate U-tube system. In operation the double hoop was filled with toluene, and the U-tube was filled with mercury. A small glass capillary tube was affixed to the open end of the U-tube, and a platinum wire was inserted in the capillary tube to serve as one electrical contact. The walls of the regulator formed the other contact, and the mercury served to connect the two. It was found after an extended period of experimentation, however, that long-term temperature stability could not be achieved with a stainless steel regulator of this type. Despite extreme efforts to insure the absence of leaks and the dimensional stability of the device, it was not possible to reduce the change in the mean temperature of the oil to less than a steady rise of 0.00025°C per day. As this change resulted in an increase in temperature of 0.01°C every forty days, the stainless steel regulator was judged to be impractical for the intended purpose and was discarded. In its stead a pyrex regulator was fabricated, the design of which was based on that of the regulators used for the same purpose at NBS, Washington, and at the present time both oil baths are controlled by pyrex regulators.

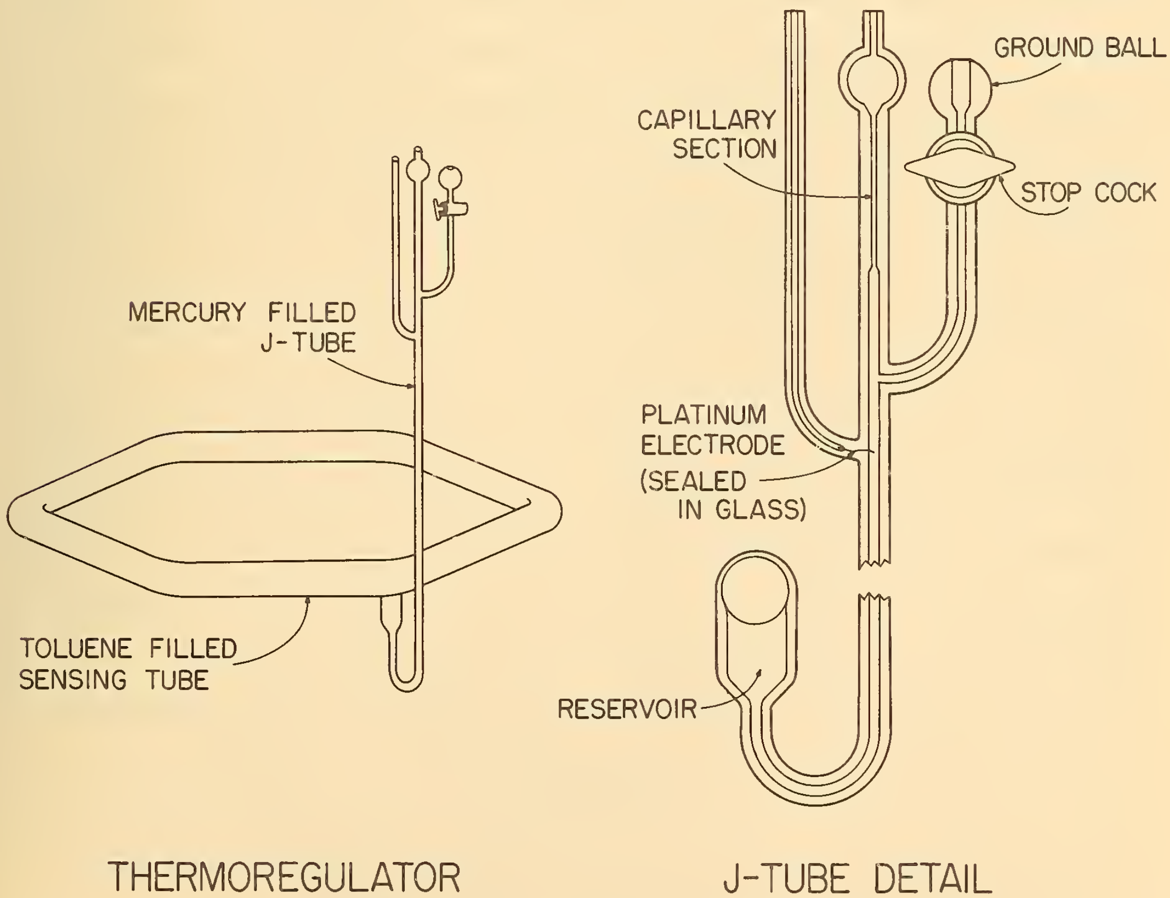

FIGURE 9. PYREX THERMOREGULATOR

The pyrex regulator consists of a one-inch o. d. glass tube, bent into the approximate form of a toroid. Sealed to this sensing tube is a small tube bent in the form of a J (figure 9). In operation, the sensing tube is filled with toluene, and the J-tube is filled with mercury. As in the stainless steel regulator, the mercury serves both to contain the toluene and to provide an electrical path between two electrodes. The upper, open end of the J-tube is drawn down to about 0. 6 mm, i. d., and it is in this section that the platinum wire is inserted. The small i. d. of this section serves to amplify the changes in the height of the mercury column resulting from the thermal expansion and contraction of the toluene. At the top of the capillary section is a bulb which serves as an overflow chamber. The J-tube also has two other tubes sealed to it somewhat below the capillary section. One of these contains a platinum electrode sealed in the glass at the junction between the two tubes. This electrode, the platinum wire inserted in the capillary tube, and the mercury column comprise the control switch of the system. The second tube that is sealed to the J-tube is used for filling the regulator and for adding or removing mercury when adjusting the control temperature and contains a stopcock to facilitate opening the tube for these purposes. The J-tube is enlarged in diameter for a short distance from the junction between it and the sensing tube. This enlarged space serves as a reservoir for mercury. Obviously great care must be exercised to prevent the possibility of air remaining in the sensing tube in order to insure proper operation of the regulator since the presence of air manifests itself in two undesirable ways: it causes the action of the regulator to be erratic rather than periodic, and it causes the temperature of the oil to vary with changes in atmospheric pressure. These effects are illustrated in figure 10. Therefore, before the toluene was introduced into the regulator it was boiled for a few minutes to insure that all dissolved air was removed.[4]

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[4] When boiling toluene appropriate safety measures must be observed as toluene is highly flammable.

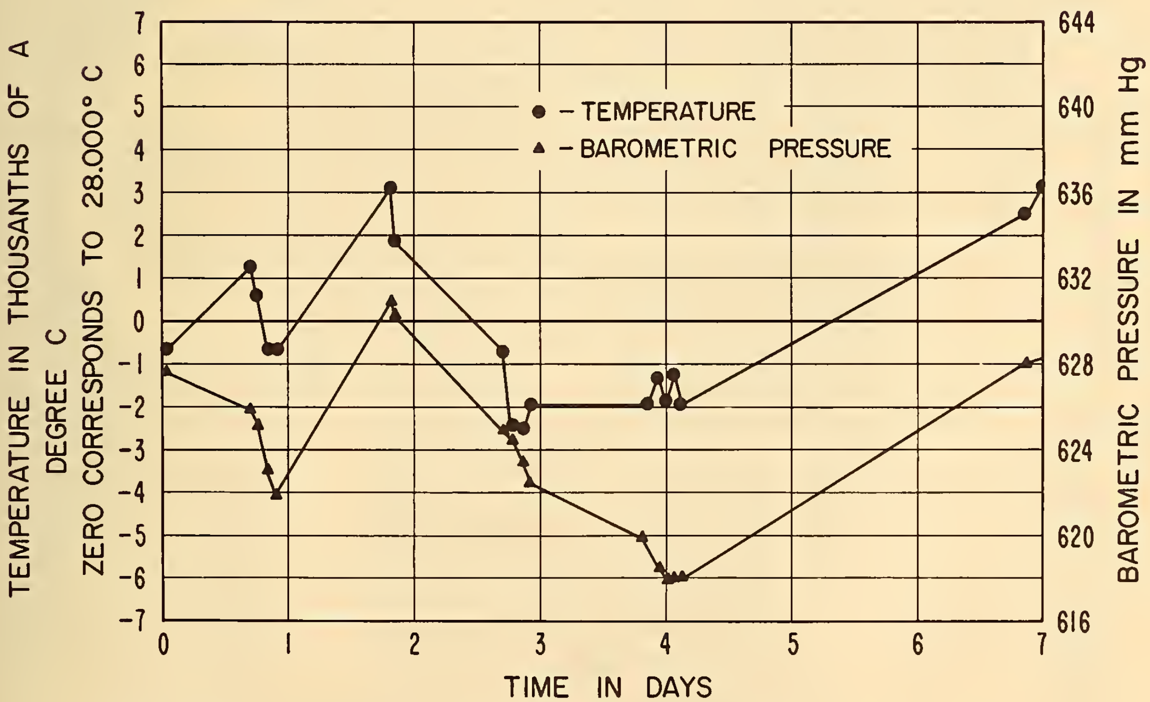

FIGURE 10. RELATIONSHIP OF TEMPERATURE AND ATMOSPHERIC PRESSURE RESULTING FROM ENTRAPPED AIR IN THERMOREGULATOR

When filling the regulator with toluene the following procedure was carried out. The top of the capillary tube was sealed, and a flexible tube was connected to the arm containing the stopcock. The other end of this tube was connected to one opening of a three-way valve. A second opening was connected to a vacuum pump, and the third was connected to a flask containing the freshly boiled toluene. A vacuum was then drawn on the regulator, and, by means of the three-way valve, the toluene was allowed to fill the regulator without contaminating the vacuum pump. The apparatus used in filling the regulator is shown in figure 11. Even with the precautions noted above, however, a small bubble remained when the toluene ceased to flow into the regulator. This bubble was then carefully removed by the following means. The flask of toluene was connected to the regulator by a flexible tube. The regulator was then cooled by packing ice and water around the sensing tube. After it had been allowed to cool for some time it was carefully overturned to trap the bubble in the mercury reservoir chamber. The regulator was then gently warmed by holding the sensing tube with bare hands, and the expansion of the toluene forced the bubble out of the tube. The regulator was then brought to a temperature approximately one degree higher than the highest temperature at which it would be used to regulate, and the J-tube was filled with mercury. The regulator was allowed to cool slowly, and mercury was continually added to replace that drawn into the reservoir by the contracting toluene. Thus the J-tube was filled with mercury with much less danger to the regulator than that existing when an attempt was made to fill the tube while gently tapping it to jar the mercury into place. Because of the fragility of the thermoregulator an acrylic plastic cover has been placed over that part which extends above the oil.

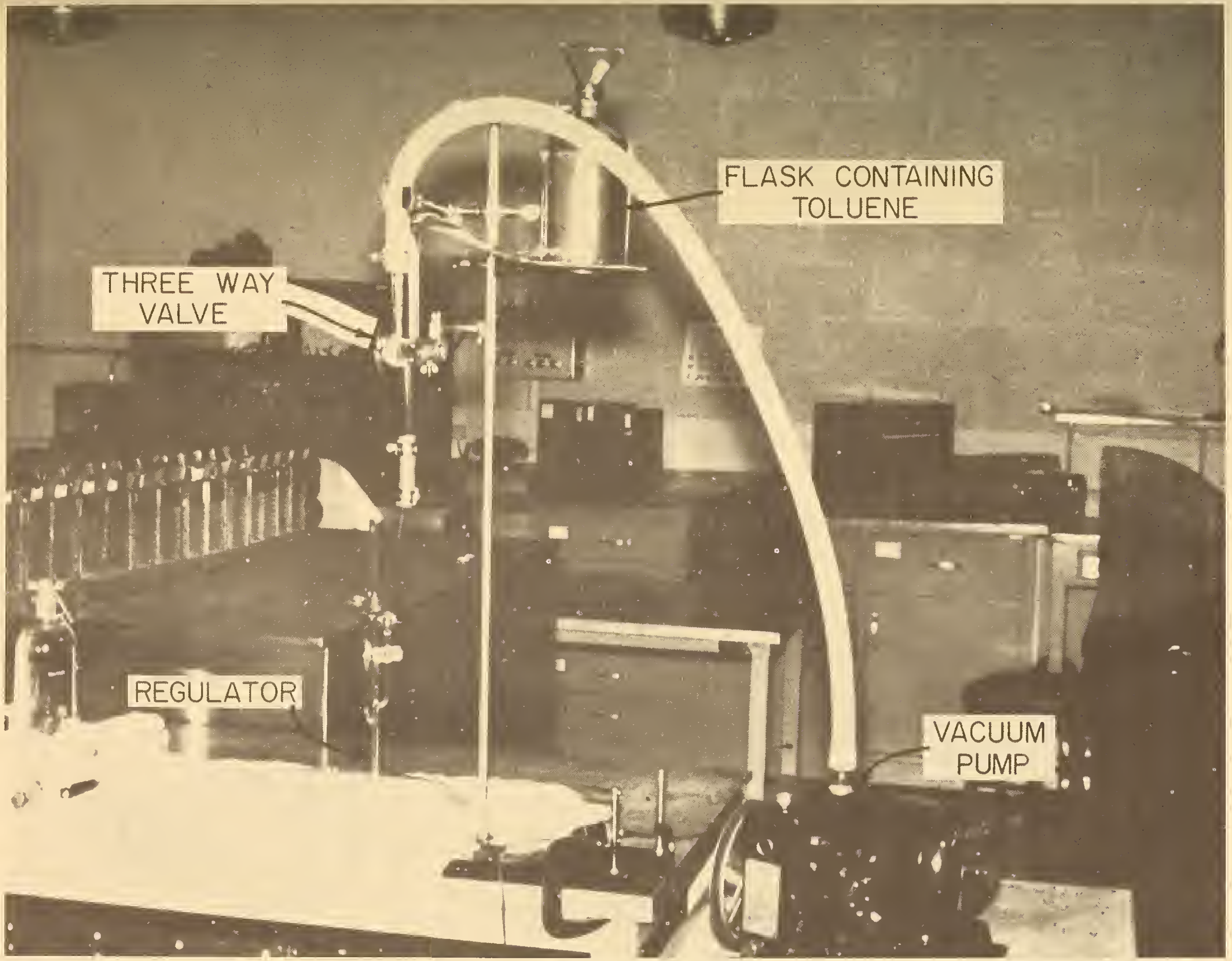

FIGURE 11. APPARATUS USED IN FILLING THE THERMOREGULATOR

3. 6 Heater Control System

The thermoregulator operates a sensitive relay which in turn operates a singlepole, double-throw relay connecting one of two variable transformers to the heater. A schematic diagram is shown in figure 12. By means of the variable transformers the current in the heater can be adjusted so that in the high-heat condition it has a value slightly above that needed to maintain the desired temperature and in the low-heat condition has a value slightly below that needed to maintain temperature. Overshoot is greatly reduced by use of a separate heater, hereafter referred to as the anticipator, which consists of 30 ohms of No. 22 Nichrome wire wound around the sensing tube of the thermoregulator.[5] Although the main heater is constantly supplied with current as explained above, the anticipator operates on an on-off cycle, i. e., there is current in it during the high-heat interval but no current in it during the low-heat interval. The anticipator thus causes a larger change in temperature to occur in the thermoregulator than in the oil, and the thermoregulator also detects this temperature change much sooner than it would if the anticipator were not used. As a result, the current in the main heater is reduced sooner than it would be without the operation of the anticipator, and overshoot is minimized. The control circuits are contained in a space below the drain and pre-heater tanks (figure 13). All relays are of the plug-in type to facilitate quick replacement. Figure 14 shows a close view of the relay chassis. During the testing of the bath it was noticed that the action of the thermoregulator caused excessive chattering of the relays. The chattering was eliminated by the addition of an RC delay circuit connected in the sensitive relay coil circuit. The relay is now delayed about one-fifth second on closing and about one-half second on opening.

[5] The use of an anticipating circuit was suggested by a regulating system designed by T. Deighton [11].

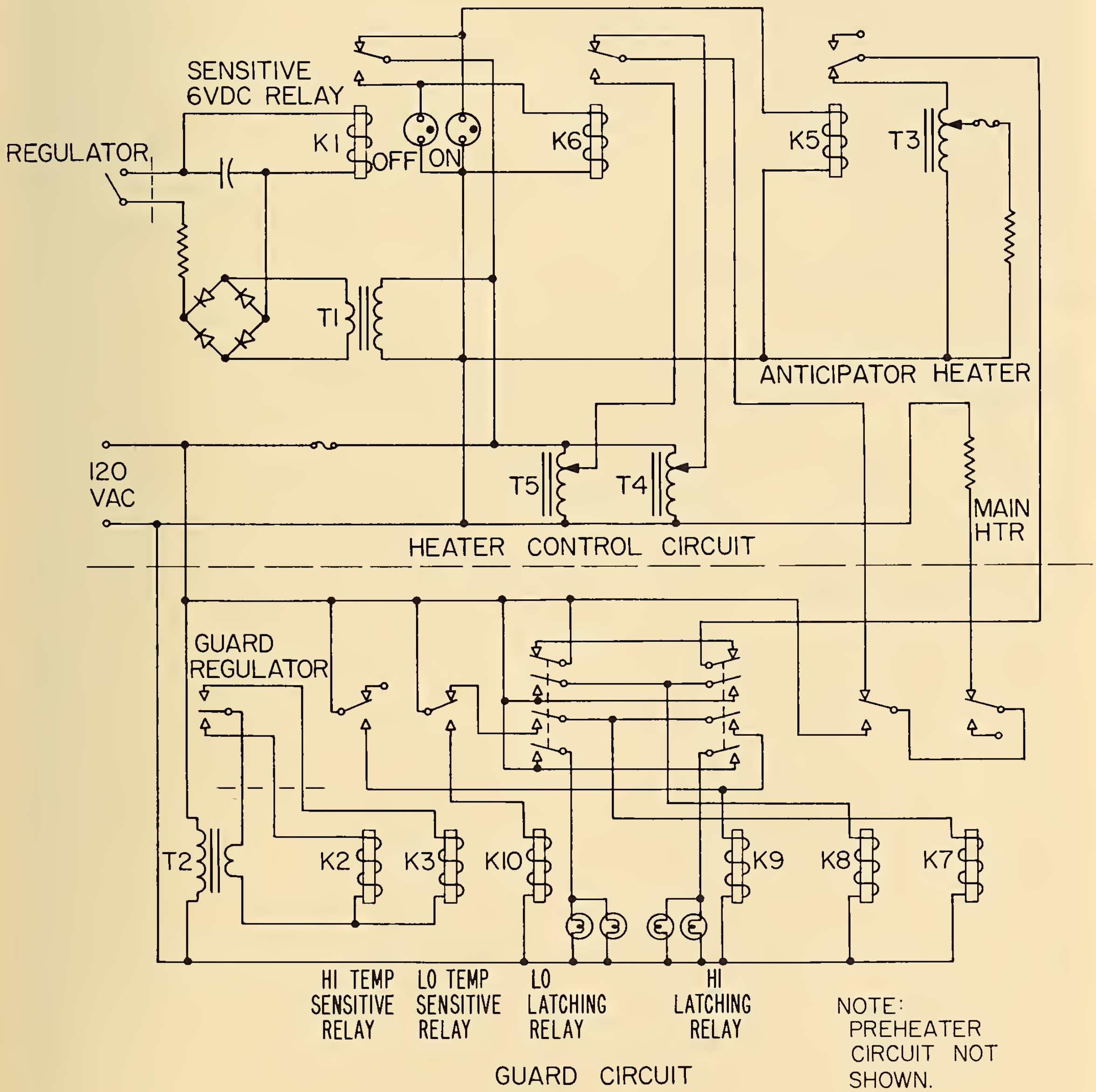

FIGURE 12. SCHEMATIC DIAGRAM OF CONTROL CIRCUITS

FIGURE 13. CONTROL CIRCUITS SECTION

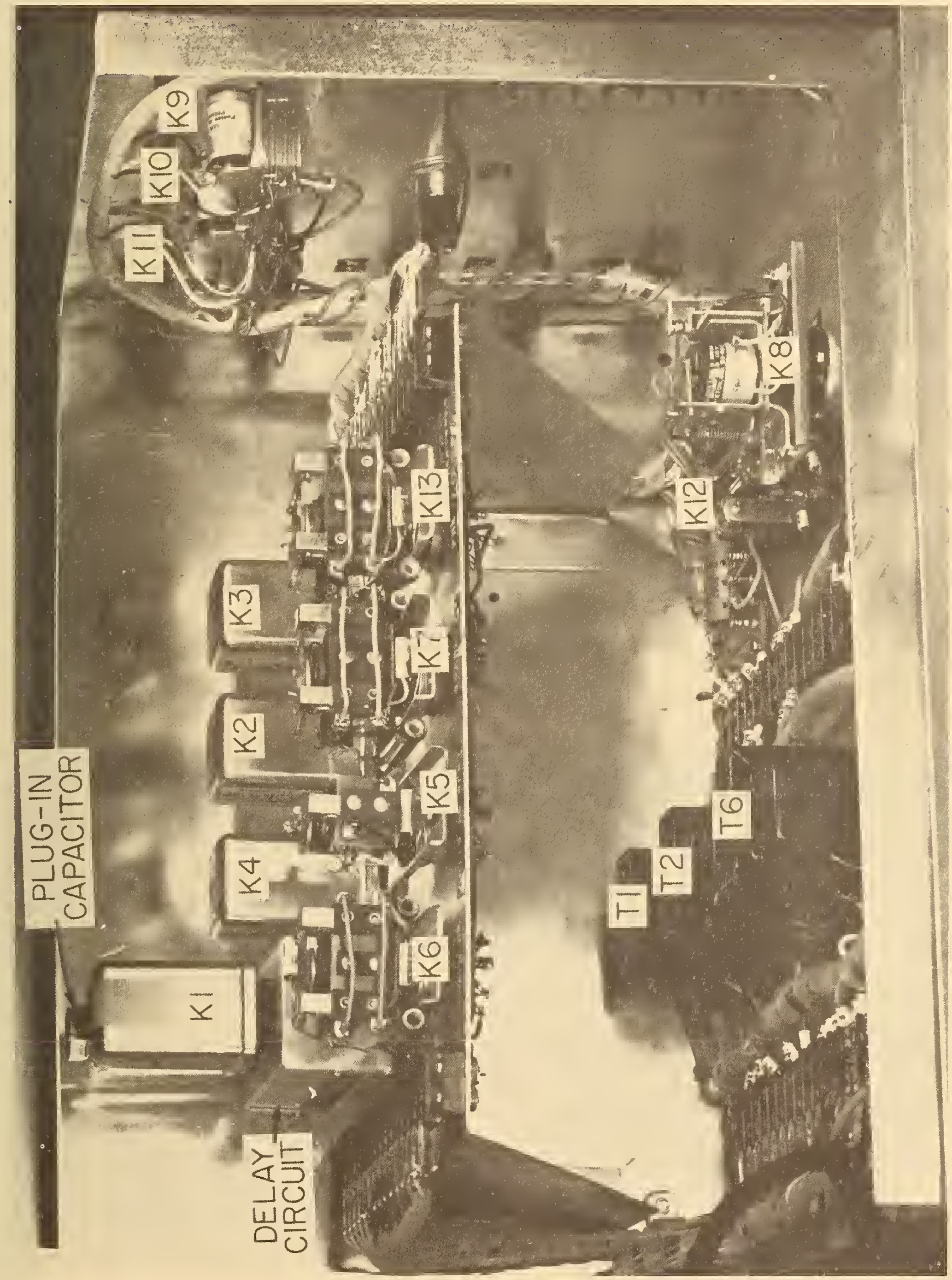

FIGURE 14. RELAY CHASSIS

3.7 Guard Circuit

As the temperature of standard cells must be kept below about 40°C [3] to [8], provision must be made in any cell enclosure to insure that malfunction of the heater control circuit does not cause the temperature to exceed this value. In addition, it was felt that even under emergency conditions, in order to minimize temperature shock to the cells, the temperature of the baths should not be allowed to vary more than one degree Celsius. With these factors in mind a guard circuit (figure 12) was built into the bath. This circuit will completely disconnect the regulator and heater system and connect the heater through the guard thermoregulator to 120 volts a-c and associated relays. Thus, if for any reason the temperature of the bath either increases or decreases by about one degree, the guard circuit will take over and start regulating to about ±0.1°C at the new temperature. In addition to the circuitry in the baths, an emergency power generator has been installed which supplies power to the standard cell room in the event of failure of normal power. The guard circuit has been designed to remain in control, once it has taken over control, until manually disconnected. This was done to insure a positive indication of large changes in temperature that would otherwise be noticed only by the behavior of the standard cells. The guard regulator is of the bi-metal type.

3.8 Electrical Connections to Cells

In order to minimize thermal electromotive forces, copper-to-copper connections are used in all cell emf circuits. Originally an attempt was made to eliminate any junctions in the wires from the cell groups to the potentiometer. However, this proved to be very complex and inconvenient, and a study was made of the effects of copper-to-copper connections. The conclusion was reached that connections of this type do not produce measurable thermal electromotive forces. As a further precaution the positive and negative leads were twisted together as tightly as the insulation would permit to minimize the effects of the Thomson emf. The connections to the cell groups are made by methods appropriate to the several forms of cell racks. In the most common form the cell leads are brought out to copper pins at the top of the rack and for this type a special clamp was designed which is shown in figure 15.

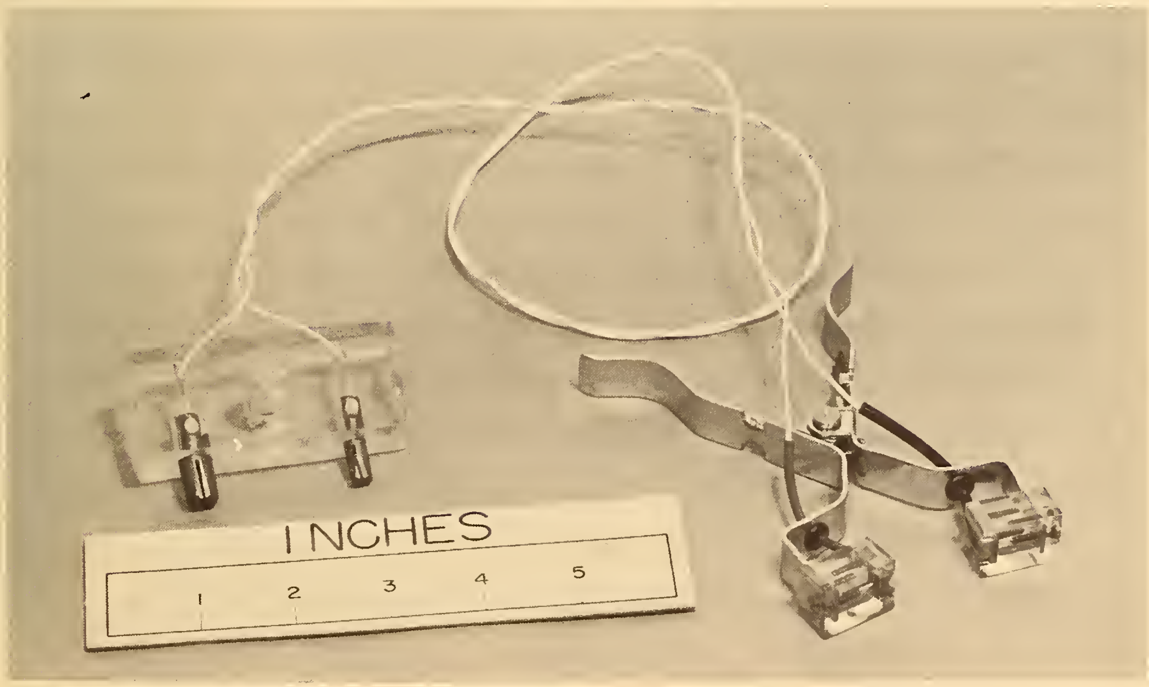

FIGURE 15. CONTACT CLAMP

3.9 Pre-heater and Drain Tanks

A small pre-heater tank (figure 5) was incorporated into the design of the 28°C bath for use when adding groups of cells to the bath. Since the NBS groups are contained in this bath, it was suggested that the introduction of cold (room temperature) groups into the oil would be undesirable in that it would reduce its temperature temporarily and also cause temperature gradients. This was found to be the case. The effects last an hour or two after the new group has been placed. The inclusion of the pre-heater provides for bringing the group and its basket to 28°C ± 0.2°C before immersion in the main tank and, as a result, the emf of the reference group remains undisturbed by the introduction of new cells. Thus the calibration of cells is not interrupted. A pre-heater was also incorporated in the 35°C bath to provide an effective increase in its capacity. As cells must be kept at temperature for four to six weeks before they can be calibrated, it was felt that the capacity of the bath could be considerably increased by providing a second tank in which the cells could spend the first two or three weeks of this waiting period. As the temperature requirements on the pre-heater are much less stringent than on the main tank, an adjustable, mercury-in-glass thermoregulator that is commercially available was used in a simple on-off circuit. The pre-heater in the 35° bath can contain up to ten groups of cells and thus effectively increase the capacity of the bath to 22 groups. The drain tanks (figure 5) were incorporated into the baths to reduce the possibility of spillage of oil on the floor and on the operator. The cells may be left in either drain tank for an extended period as the drain tanks also are protected from light.

4. Performance

The 28°C bath has been in operation for over a year, and the 35°C bath has been in operation for about eight months. At first and immediately following any drastic temperature adjustment (the 35°C bath was adjusted to 28°C for a short period), a slow erratic rise in temperature was noted as shown in figure 16. This evidently was due to the escape of small bubbles of entrapped air in the mercury column. After some time, however, this change leveled off after a total rise of about 0.02°C. At present the cyclic change in temperature is less than ± 0.001°C. This results in a variation in cell emf of no greater than 0.1 microvolt. The day-to-day change in mean temperature does not exceed 0.002°C. As can be seen in figure 17, the temperature is still climbing very slowly - roughly 0.001°C every 20 days. It has been found that the mercury column of the thermoregulator must be cleaned two or three times a year to remove any foreign material from the top of the column in the capillary tube. If this is not done, the platinum wire will become insulated by the material, and the temperature will suddenly rise by as much as 0.01°C. Even after this occurs, however, the normal state can be reattained by cleaning the column and the wire.

As a result of the incorporation of these oil baths into the standard cell unit at the Boulder Laboratories, fluctuations in cell emf due to variations in temperature have been reduced to such an extent that the accuracy with which the volt is maintained at the Boulder Laboratories is no longer limited by temperature variations.

5. Suggestions for Improvement

Although the present design objectives were attained by the presently operating baths, it is believed that the convenience of temperature adjustment and system reliability could be further improved by the replacement of the mercury-toluene regulator and relay system with an all solid state electronic system. The primary difficulty with present electronic systems is the lack of long term stability and, in the case of vacuum tube systems, poor reliability. The solution of the stability problem coupled with the reliability associated with the use of solid state devices could lead to an improvement over the present system. In the event that such an electronic control is perfected, it will be incorporated in the present baths.

The author is indebted to Mr. Frank D. Weaver for his inspiration and assistance during the design of the baths at the Boulder Laboratories, and also to Dr. Walter J. Hamer and Miss Catherine Law of the Electrochemistry Section of NBS Washington for their help.

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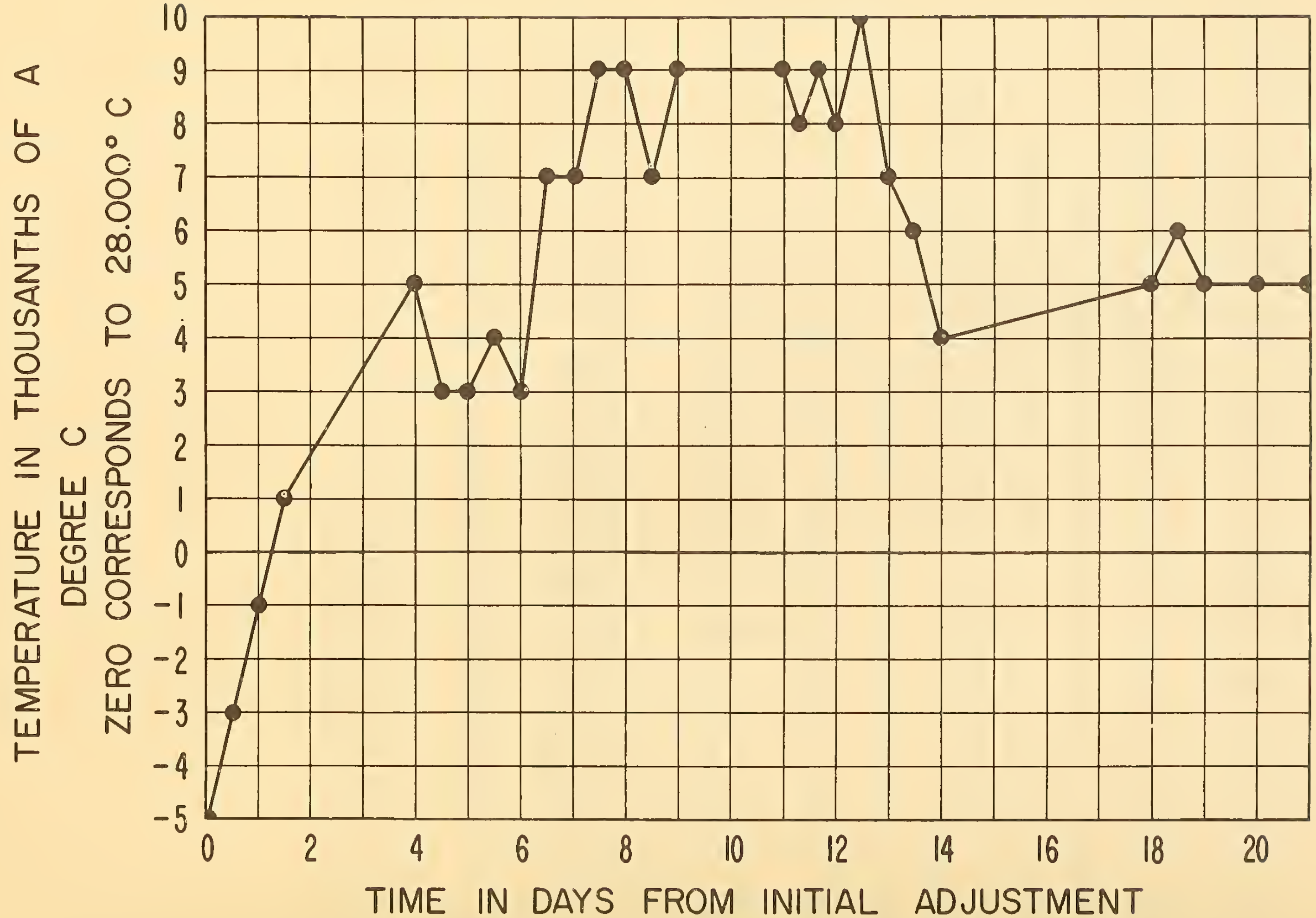

FIGURE 16. TEMPERATURE VARIATIONS FOLLOWING INITIAL ADJUSTMENT OF TEMPERATURE IN 28°C OIL BATH

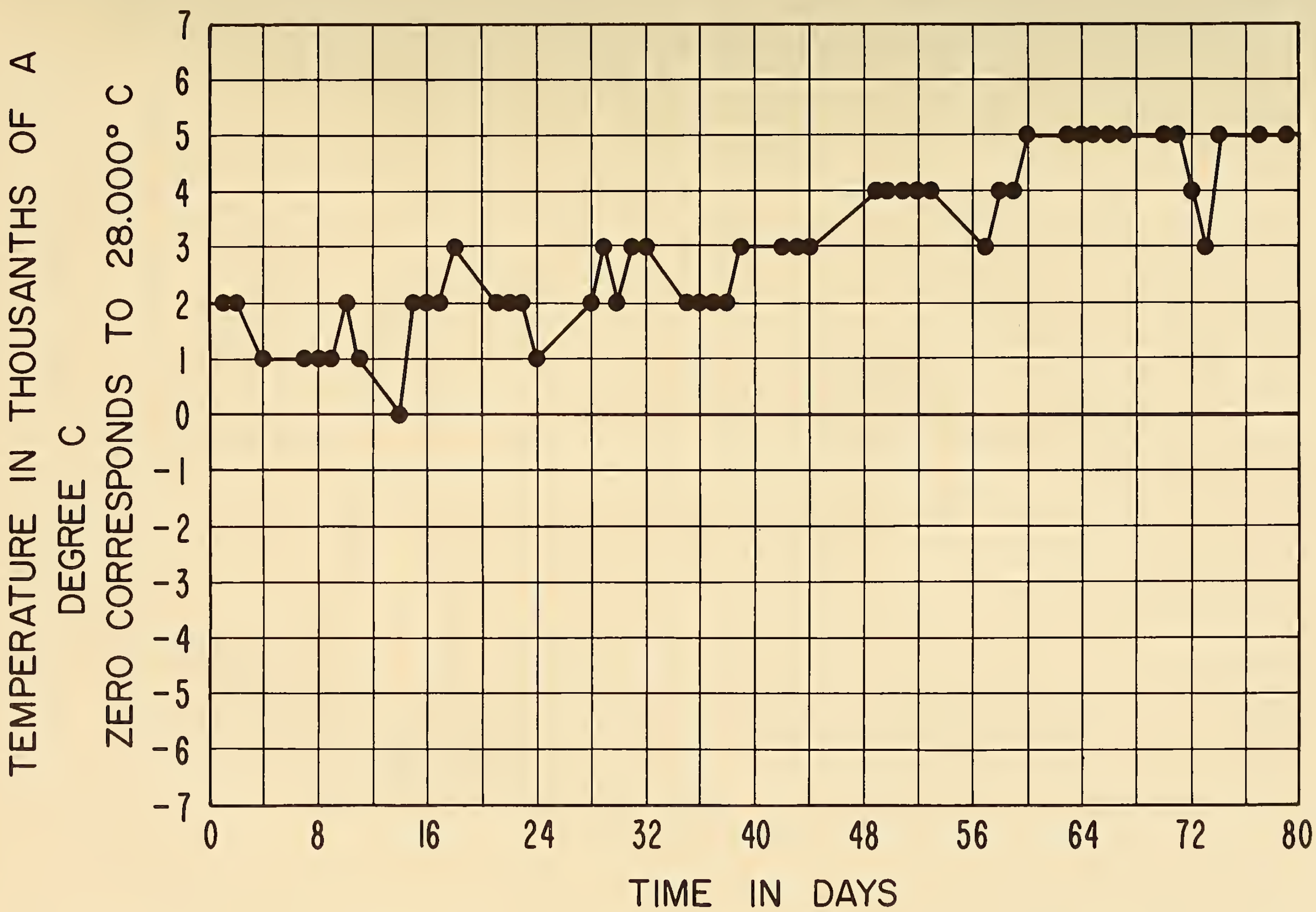

FIGURE 17. DAY-TO-DAY TEMPERATURE VARIATIONS OVER AN 80 DAY PERIOD

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U. S. DEPARTMENT OF COMMERCE
Luther H. Hodges, *Secretary*

NATIONAL BUREAU OF STANDARDS
A. V. Astin, *Director*

THE NATIONAL BUREAU OF STANDARDS

The scope of activities of the National Bureau of Standards at its major laboratories in Washington, D.C., and Boulder, Colorado, is suggested in the following listing of the divisions and sections engaged in technical work. In general, each section carries out specialized research, development, and engineering in the field indicated by its title. A brief description of the activities, and of the resultant publications, appears on the inside of the front cover.

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Electricity. Resistance and Reactance. Electrochemistry. Electrical Instruments. Magnetic Measurements. Dielectrics. High Voltage.

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Instrumentation. Engineering Electronics. Electron Devices. Electronic Instrumentation. Mechanical Instruments. Basic Instrumentation.

Physical Chemistry. Thermochemistry. Surface Chemistry. Organic Chemistry. Molecular Spectroscopy. Molecular Kinetics. Mass Spectrometry.

Office of Weights and Measures.

BOULDER, COLO.

Cryogenic Engineering Laboratory. Cryogenic Equipment. Cryogenic Processes. Properties of Materials. Cryogenic Technical Services.

CENTRAL RADIO PROPAGATION LABORATORY

Ionosphere Research and Propagation. Low Frequency and Very Low Frequency Research. Ionosphere Research. Prediction Services. Sun-Earth Relationships. Field Engineering. Radio Warning Services. Vertical Soundings Research.

Radio Propagation Engineering. Data Reduction Instrumentation. Radio Noise. Tropospheric Measurements. Tropospheric Analysis. Propagation-Terrain Effects. Radio-Meteorology. Lower Atmosphere Physics.

Radio Systems. Applied Electromagnetic Theory. High Frequency and Very High Frequency Research. Modulation Research. Antenna Research. Navigation Systems.

Upper Atmosphere and Space Physics. Upper Atmosphere and Plasma Physics. Ionosphere and Exosphere Scatter. Airglow and Aurora. Ionospheric Radio Astronomy.

RADIO STANDARDS LABORATORY

Radio Physics. Radio Broadcast Service. Radio and Microwave Materials. Atomic Frequency and Time-Interval Standards. Millimeter-Wave Research.

Circuit Standards. High Frequency Electrical Standards. Microwave Circuit Standards. Electronic Calibration Center.

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