JOURNAL OF RESEARCH of the National Bureau of Standards—C. Engineering and Instrumentation
Vol. 66C, No. 3, July–September 1962

A Furnace for Thermocouple Calibrations to 2,200 °C

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(February 26, 1962)

A tantalum-tube furnace has been constructed to calibrate and investigate the thermoelectric behavior of high-temperature thermocouples. The furnace and its associated equipment were designed with emphasis on features that would assure a high degree of accuracy in measurements that are made at high temperatures and also with emphasis on trouble-free performance. Data that were obtained during furnace operation showed that thermocouple depth of immersion into a properly designed blackbody is of considerable importance if good agreement is to be realized between a calibrated optical pyrometer and a calibrated thermocouple that has been placed in the hot zone of the furnace. High-purity helium gas can be used in the furnace to keep thermocouple contamination to a minimum.

1. Introduction

Furnaces consisting of a single metal tube which is heated to temperatures as high as 3,000 °C have been reported in the literature [1, 2, 3, 4, 5].[1] Although all of these furnaces have the same basic design, each has minor modifications that adapt it to a particular type of high-temperature work. Nadler, Runyan, and Kempter [1], for example, designed a furnace with emphasis on its ability to operate in a vacuum or in an inert or reducing gas up to a pressure of 100 atm. The furnaces described by Sims, Gaines, and Jaffee [4] and by Lachman and Kuether [5] were designed specifically for thermocouple calibrations.

Some of the features that are essential in the design of a high temperature[2] thermocouple calibrating furnace are, (1) The thermocouple should be freely suspended in the hot zone of the furnace. Any obstructions along the entrance path into the furnace would require undesirable bending of the thermocouple wires and could conduct heat away from the wires which might result in an erroneous calibration. Also, any materials that are placed in contact with the thermocouple at high temperatures could bring about solid state reactions between the thermoelements and these materials with resulting local diffusional contamination of the thermoelements. (2) The measuring junction of the thermocouple should be contained in a blackbody enclosure with provisions for viewing the radiation emanating from the enclosure. This arrangement is essential since the International Practical Temperature Scale of 1948 above 1,063 °C is defined in terms of blackbody radiation [6]. If this blackbody radiation is observed with a calibrated optical pyrometer [7], the temperature of the measuring junction of the thermocouple can be accurately determined. However, if the radiation in the vicinity of the thermocouple measuring junction is not blackbody radiation, an emissivity correction must be applied to the optical pyrometer readings. In many cases, emissivity corrections cannot be accurately determined. (3) The temperature profile along the thermocouple wires should be as follows: The measuring junction and a specific length[3] of the thermocouple wires leading directly from the junction should be in a region of uniform temperature. Without this temperature uniformity, it is likely that a significant amount of heat will be conducted away from the measuring junction along the thermocouple wires thus causing the thermocouple to indicate a temperature which is lower than its environment. From this uniform temperature region to the water cooled shell of the furnace, the temperature should decrease uniformly with no sharp temperature gradients existing at or near the thermocouple wires. Freeman [8] observed significantly large differences in the emf developed by thermocouples when the thermocouple wires were subjected to extreme gradients from the emf developed by the same thermocouples under normal gradient conditions. (4) At temperatures in the 2,000 °C region (and higher), a furnace should be designed to allow the thermocouple to be suspended free from any electrically insulating or supporting materials in the hot zone. The electrical resistivity of known available materials in this temperature range becomes quite low and can give rise to a short circuiting effect between the thermocouple wires. (5) The furnace design should be such that all internal parts can be easily out-gassed or baked out in order to maintain a high vacuum or clean atmosphere during calibration runs. If calibrations are performed in an inert atmosphere, the inert gas that is used should be of research grade quality. Some inert gases of "commercial grade" quality have relatively large percentages of impurities and these impurities can have a serious effect on the physical and chemical properties of a thermocouple at high temperatures. Refractory oxides should be eliminated from the interior of the furnace wherever possible since such materials usually continue to out-gas for long periods of time. (6) The thermal inertia of the furnace

[1] Figures in brackets indicate the literature references at the end of this paper.
[2] High temperature meaning between 1,000 and 3,000 °C.

[3] The length of the thermocouple wires that should be at a uniform temperature depends primarily upon the diameter of the wires. The length that is needed for 0.020 in. diam wires is discussed later in this paper.

should be such that any desired temperature can be reached in a relatively short time. When a desired temperature has been reached, thermal stability throughout the furnace should prevail shortly thereafter.

2. Furnace

A high temperature tantalum tube furnace has been built at the National Bureau of Standards to investigate and calibrate thermocouples and thermocouple materials at temperatures up to 2,200 °C. In constructing the furnace considerable emphasis was placed on a design that would entail all of the above mentioned features. The heater in the furnace is a seamless tantalum tube[4] with an outside diameter of 1.0 in., a wall thickness of 0.020 in. and an overall length of 18.25 in. Its electrical resistance is 0.0022 ohm at room temperature and 0.0126 ohm at 2,200 °C. A thermal expansion joint (15, fig. 1) was made in the upper section of the tube by cutting the tube lengthwise into six sections and then spreading the sections to form wedge-shaped "arms." Both the upper and lower tantalum rings (14 and 32, fig. 1) were "electron beam" welded to the ends of the tantalum tube. The lower tantalum ring is bolted to a water-cooled square copper plate (33, fig. 1) which contains an O-ring gasket on the underside. By removing the bottom plate bolt (34, fig. 1), the tantalum tube and top plate can be removed from the furnace shell.

In the initial design of the furnace, it was calculated that blackbody conditions would prevail inside of the tantalum tube at the level of the sighting hole (28, fig. 1) provided that a tantalum tube was selected with proper dimensions. In order to evaluate this calculation a calibrated Pt 6 percent Rh versus Pt 30 percent Rh thermocouple was placed inside of the tantalum tube with its measuring junction at the level of the sighting hole. A standard Pt versus Pt 10 percent Rh thermocouple was not used in this instance since the platinum element showed various degrees of instability which was probably due to tantalum or molybdenum vapor contamination. A considerable amount of data was taken at temperatures in the 1,000 to 1,200 °C range in which the calibrated thermocouple emf readings and the calibrated optical pyrometer[5] readings were made simultaneously. The data showed that the temperature indicated by the thermocouple was from 4 to 5 deg lower than the temperature indicated by the optical pyrometer. Comparisons between the calibrated thermocouple and the optical pyrometer at temperatures above 1,200 °C were not made since there was no readily available method for calibrating a thermocouple above this temperature to an accuracy better than ± 1.0 deg C. A good agreement between the thermocouple and the optical pyrometer in a furnace of this type is extremely important at lower temperatures (1,000 to 1,200 °C) since a discrepancy of 4 or 5 deg C may increase to 10 or 15 deg C at temperatures in the 2,000 °C region.

To make certain that this 4 to 5 deg C discrepancy was not due to calibration errors in the optical pyrometer, a careful calibration check was made between the optical pyrometer in question and the Fairchild optical pyrometer[6] at NBS. About a half dozen observations were made by each of several experienced observers on both instruments and the average of their observations showed that the two optical pyrometers agreed within 0.2 deg at 1,170 °C. This eliminated the possibility of a sufficiently large calibration error in the optical pyrometer in question. In addition, the Pt 6 percent Rh versus Pt 30 percent Rh thermocouple that was used was recalibrated to determine whether the discrepancy could have resulted from a calibration change in the thermocouple. The second calibration indicated a thermocouple emf change of less than 0.3 deg at 1,100 °C. It was therefore concluded, contrary to initial calculations, that either blackbody conditions did not prevail inside of the tantalum tube or that the thermocouple indicated a lower temperature because of heat loss upward from the thermocouple measuring junction.

In order to resolve this discrepancy, it was felt that a separate blackbody cavity contained within the central portion of the tantalum tube might be a possible solution to the problem. This blackbody cavity would provide a longer isothermal region in the vicinity of the thermocouple measuring junction. Blackbody cavities of various shapes and dimensions were fabricated and placed inside of the tantalum tube for evaluation in the 1,000 to 1,200 °C range. A molybdenum blackbody (fig. 2) in the shape of a cylinder crucible containing a thorium oxide lid was found to bring about the best agreement between the thermocouple and the optical pyrometer. The thorium oxide lid serves to electrically insulate the thermocouple wires from the walls of the molybdenum blackbody at low furnace temperatures. As it was stated in the introduction, the resistivity of insulating materials becomes quite low at temperatures above 2,000 °C and thorium oxide is not an exception. However, if the thermocouple wires come in contact with the thorium oxide lid at temperatures above 2,000 °C, there is an immediate indication of a partially rectified a-c voltage[7] on the galvanometer in the emf measuring circuit. When this occurs the thermocouple wires can be manually repositioned (through the thermocouple wire seals) until the wires hang freely in the center hole of the thorium oxide lid. The molybdenum blackbody is supported inside of the tantalum tube by four tungsten wires (0.030 in. diam) that are attached to the top plate of the furnace. The uniqueness of this design is that as the

[4] The tube was obtained from the Fansteel Metallurgical Corporation, North Chicago, Ill.

[5] The optical pyrometer used in this work was a modified commercial optical pyrometer that was calibrated on a basis of temperature versus optical pyrometer lamp current. A higher accuracy can often be realized with an optical pyrometer calibrated and used in this manner [7].

[6] The NBS Fairchild optical pyrometer, also referred to in the literature [7] as "The NBS Optical Pyrometer" is a high precision instrument designed by C. O. Fairchild of NBS and is used at NBS for testing other optical pyrometers and pyrometer lamps submitted for calibration.

[7] The a-c voltage applied to the furnace heating element is partially rectified by the thorium oxide.

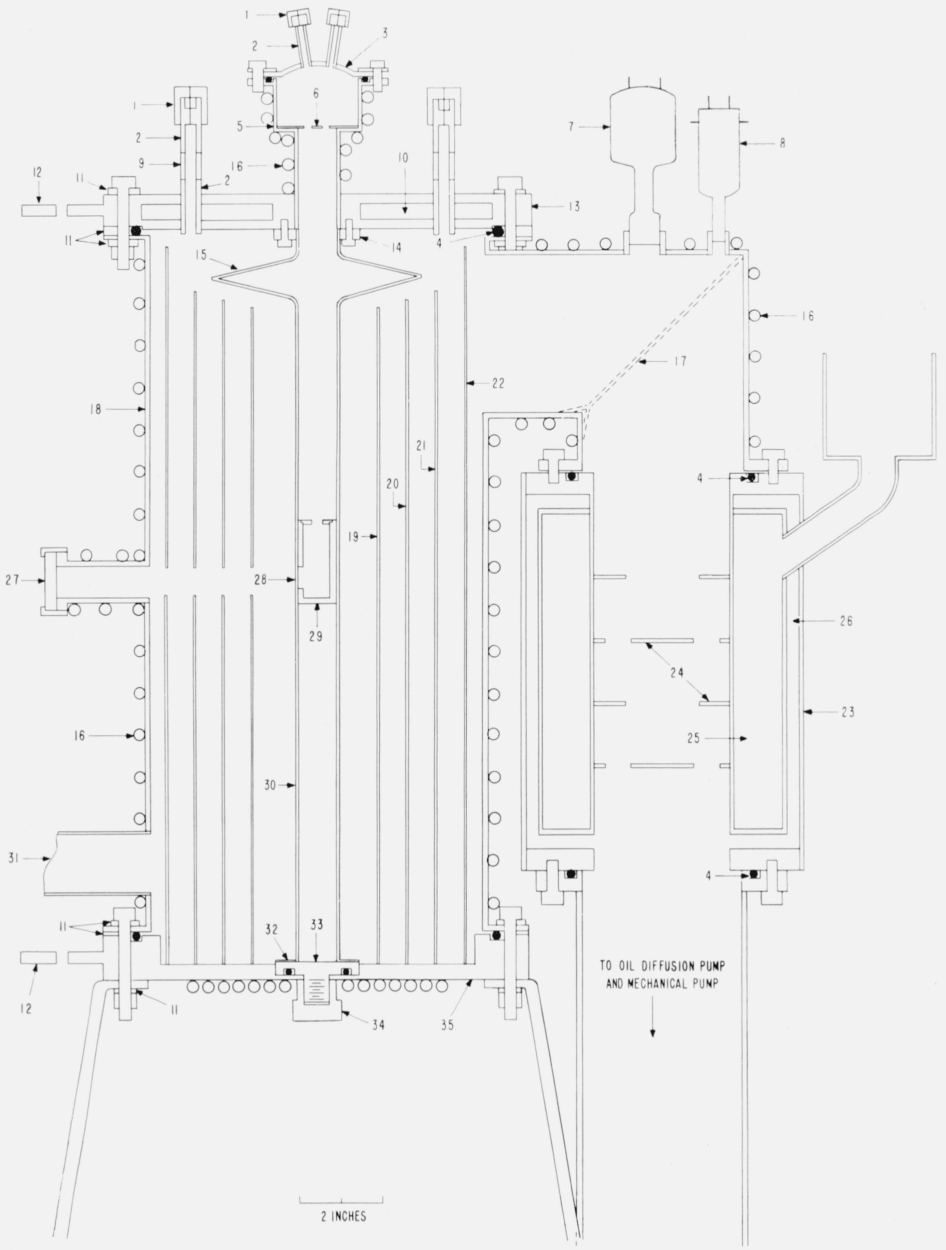

FIGURE 1. *Tantalum-tube furnace.*

1. Caps for making vacuum tight seals around thermocouple wires. A cylinder type neoprene gasket is compressed around thermocouple wires.
2. Kovar metal tube.
3. Dome made of #7052 glass providing electrical insulation for thermocouple elements.
4. Neoprene O-ring gasket.
5. Top plate extension (brass).
6. Aluminum oxide radiation shield.
7. Ionization vacuum gage.
8. Thermocouple vacuum gage.
9. 7052 glass tube providing electrical insulation for thermocouple elements.
10. Chamber for water flow during furnace operation.
11. Electrically insulating spacers.
12. Power supply terminal.
13. Removable top plate (brass).
14. Tantalum spacing ring providing electrical contact between top plate and tantalum tube.
15. Thermal expansion joint of tantalum tube.
16. Copper tubing for water cooling.
17. Auxiliary radiation shield.
18. Furnace shell (brass).
19. First radiation shield. 0.020 in. tantalum sheet rolled into a cylinder and secured with tantalum rivets.
20. Second radiation shield. (0.020 in. molybdenum.)
21. Third radiation shield. (0.020 in. molybdenum.)
22. Fourth radiation shield. (0.010 in. molybdenum.)
23. Liquid nitrogen trap.
24. Metal baffle plates at liquid nitrogen temperature.
25. Liquid nitrogen chamber.
26. Vacuum chamber.
27. Pyrex glass window.
28. Hole (0.045 in. diam) for sighting with optical pyrometer.
29. Molybdenum blackbody.
30. Tantalum tube.
31. Inert gas entrance.
32. Tantalum rings for electrical contact.
33. Removable copper plate for electrical contact.
34. Hex-head nut for tightening copper plate again O-ring gasket
35. Bottom plate (brass).

temperature of the furnace is increased or decreased, the thermal expansion or contraction of the tungsten support wires and of the thermocouple wires is nearly equal and thus the depth of immersion of the thermocouple into the blackbody remains nearly constant.

The data obtained with this molybdenum blackbody indicate that at least 2 in.[8] of the thermocouple at the measuring junction end must be contained within the blackbody to obtain good agreement with the optical pyrometer. With a thermocouple immersion of 2 in. into the molybdenum blackbody, the differences observed between the calibrated thermocouple and the optical pyrometer were 0.4 deg at 1,000 °C, 0.3 deg at 1,100 °C, and 0.3 deg at 1,200 °C. These figures represent an average of many observations made at the respective temperatures and are well within the combined estimated uncertainties of the thermocouple and the optical pyrometer.

A thermocouple that is to be calibrated in the furnace against the optical pyrometer is suspended from two thermocouple wire seals (1, fig. 1) directly above the tantalum tube. The thermocouple hangs freely inside of the tantalum tube with its measuring junction at the level of the sighting hole. The only material in contact with the thermocouple wires is an aluminum oxide disk (6, fig. 1) which serves as a radiation shield. The thermocouple wires are threaded through two holes in this disk. Since the aluminum oxide disk is small in size and remains relatively cool during furnace operation, its out-gassing or contaminating effects are negligible. Auxiliary thermocouple seals (1, fig. 1) are located in the top plate of the furnace. Thermocouples or thermoelements can be brought into the furnace through these seals and held mechanically on the outside of the tantalum tube near the level of the sighting hole. If thermocouples are placed outside of the tantalum tube, the three inner radiation shields (19, 20, and 21, fig. 1) are removed from the furnace and a different set of shields is used. The top plate contains a total of six auxiliary seals through which three thermocouples or six thermoelements can be brought into the furnace with all of the elements fused at one common junction. This arrangement of six auxiliary seals is useful if two test thermocouples are to be compared or calibrated against a standard thermocouple. The arrangement is also useful if six thermoelements of the same type are to be statistically compared one against another. In the latter example, the exact temperature of the junction of the thermoelements need not be known.

Thermocouples can be calibrated in the furnace either in a high vacuum or in a purified inert atmosphere. Once the internal parts of the furnace have been out-gassed and the liquid nitrogen trap is put to use, a vacuum in the order of 0.1 to 0.01 μ of mercury can be maintained when all parts of the furnace are at room temperature. When the furnace is operating at a high temperature, a vacuum of between 1 and 10 μ of mercury can be obtained by continually pumping on the system and by using the liquid nitrogen trap and oil diffusion pump. A purified inert gas can be released into the furnace chamber through the inert gas inlet (31, fig. 1).

[8] This is the case for a thermocouple wire diameter of 0.020 in.

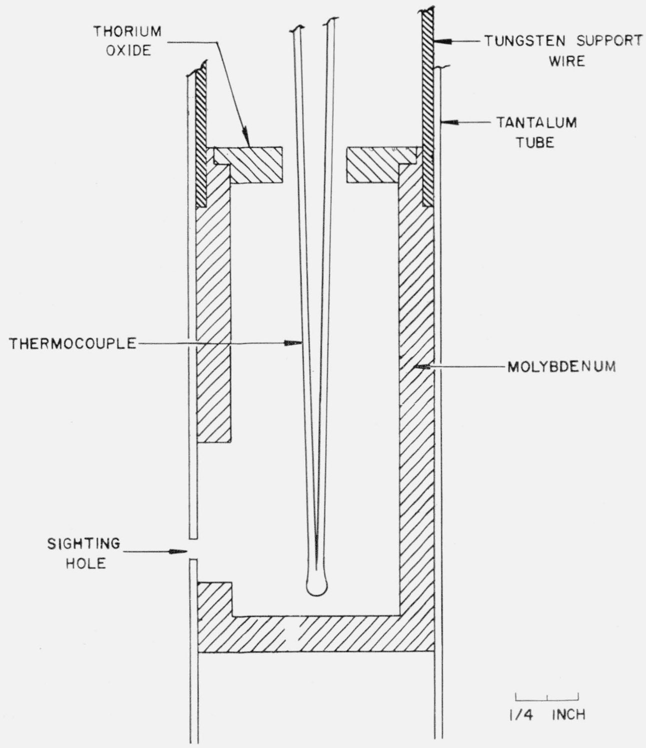

FIGURE 2. *Molybdenum blackbody.*

3. Inert Gas Purifiers

The emf developed by a thermocouple is directly related to the chemical composition and metallurgical structure of its elements. If an inert gas that is used in a thermocouple calibrating furnace contains impurities, the hot thermocouple elements may have an affinity for these impurities and consequently the chemical composition and metallurgical structure of the thermocouple may be affected. If the thermocouple is affected by these impurities, it will exhibit an asymptotic drift in emf as long as it remains in this environment. For this reason, two inert gas purifiers (fig. 3) are included as auxiliary equipment for the tantalum tube furnace. The major impurities in commercially obtained helium are oxygen, hydrogen, and nitrogen with perhaps small amounts of carbon dioxide and carbon monoxide. According to Richardson and Grant [9] large amounts of oxygen and nitrogen are absorbed by titanium metal between 800 and 1,000 °C. Likewise, zirconium metal between 300 and 400 °C will absorb large quantities of hydrogen and oxygen as reported by Gulbransen and Andrew [10]. Thus, one purifier containing titanium metal chips and one containing zirconium metal chips are used to purify the inert gas used in the furnace. The use of an inert gas of

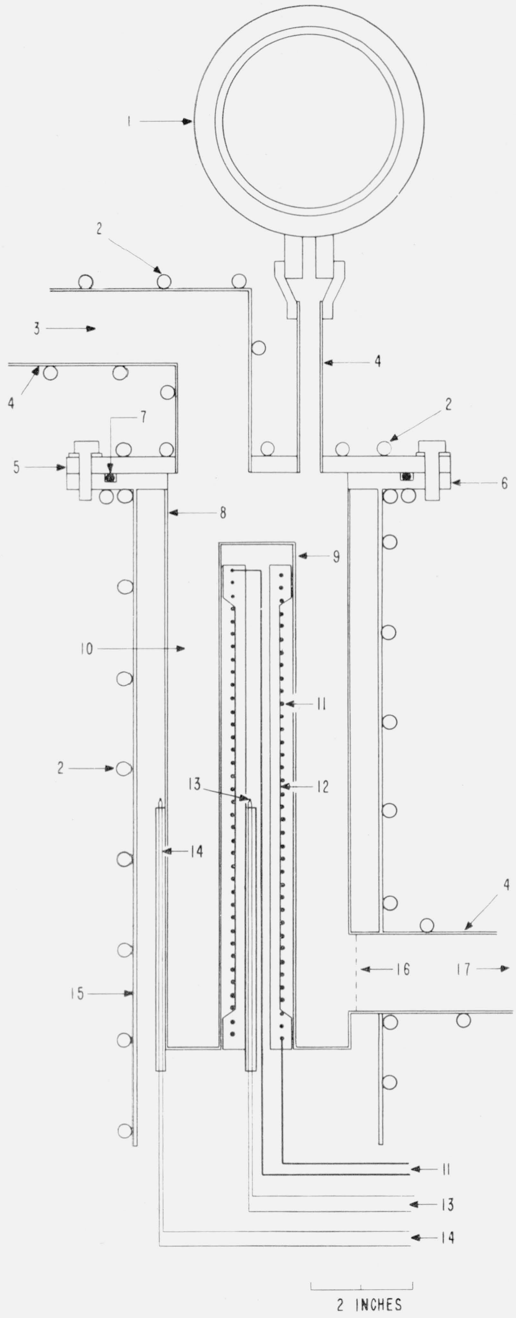

FIGURE 3. *Inert gas purifier*

1. Pressure-vacuum gage.
2. Copper water-cooling coils.
3. Inert gas entrance.
4. Stainless steel tubing.
5. Stainless steel top plate.
6. Stainless steel flange.
7. Neoprene O-ring gasket.
8. Outer wall of stainless steel tank.
9. Inner wall of stainless steel tank.
10. Chamber for titanium or zirconium chips.
11. Chromel-A 18 AWG heater winding.
12. Threaded aluminum oxide tube.
13, 14. Chromel-alumel or platinum-platinum 10 percent rhodium thermocouple.
15. Brass outer shell.
16. Stainless steel screen.
17. Inert gas exit.

high purity also serves in maintaining a longer life for the tantalum heating element. For economic reasons helium was chosen to be used in this application.

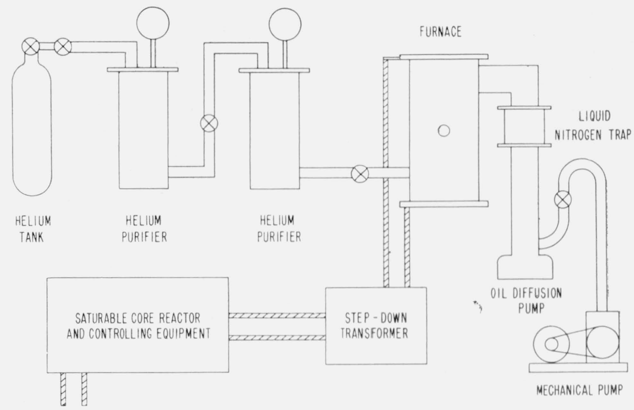

FIGURE 4. *Furnace and associated equipment.*

According to the manufacturer, the purity of the helium before it enters the two purifiers is 99.99 percent.

The two inert gas purifiers are connected in series (fig. 4) such that the gas entering the first purifier also passes through the second purifier before entering the furnace chamber. It can be seen that the inert gas is given a "double treatment" as far as oxygen removal is concerned. In most calibrations where the thermocouples are to be heated in a helium atmosphere, a quantity of helium is allowed to remain in each purifier from 3 to 6 min with the titanium and zirconium chips at optimum temperatures for the removal of impurities. In an application where high purity is not of the essence, the inert gas can flow through the purifiers at a slow rate.

4. Furnace Power Supply

Electrical power is supplied to the tantalum tube furnace through a 30 kva saturable core reactor and a step-down transformer of 12:1 ratio. Input voltage to the reactor is 200 v at 60 cycles, single phase. A modified commercial controller is used to manually select a voltage that is needed to maintain a specific furnace temperature. Once this voltage has been selected (by varying a 0 to 5 ma d-c current) the controller automatically maintains that voltage to within a close tolerance via a feedback signal from the transformer primary winding. With a stable supply voltage, the furnace temperature is held very nearly constant for reasonably long periods of time. However, since a thermal sensing device is not incorporated in this type of automatic control, it is necessary to maintain a steady flow of water to the furnace for cooling purposes. If the flow rate is not constant, noticeable temperature fluctuations may result.

During some of the initial test runs, the furnace was allowed to stabilize at a given temperature for 30 min and then the temperature fluctuations over a 10-min interval were observed with a thermocouple placed inside of the tantalum tube with the measuring junction in the molybdenum blackbody. The maximum fluctuations deduced from thermocouple readings during the 10-min interval were 0.2 deg at 1,140, 0.5 deg at 1,510, and 4.2 deg at 2,115 °C. The fluctuations at 2,115 °C were decreasing with respect to time at the end of the 10-min interval and were partly attributed to thermocouple instability resulting from inadequate annealing of the thermocouple. If the thermocouple had been allowed to anneal at the high temperature for a longer period of time, the fluctuations would have been less. Starting from room temperature, a furnace temperature of 2,200 °C can be obtained in less than 5 min. However, this fast heating rate is rarely brought about during thermocouple calibrations since a longer period of time is needed to bring about thermal stability. A power of approximately 17 kw is required to maintain a furnace temperature of 2,200 °C.

The author acknowledges the contributions made by R. J. Corruccini, National Bureau of Standards, Boulder, Colo., in the design of various parts of the furnace.

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