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Construction and Operation of a Simple High-Precision Copper-Point Blackbody and Furnace

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NBS TECHNICAL NOTE 483

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Construction and Operation of a Simple High-Precision Copper-Point Blackbody and Furnace

R. D. Lee

Detailed instructions for the construction and operation of an inexpensive copper-point blackbody and furnace are presented. Such a source may be expected to realize the radiance temperature of 1083.3°C (Int. 48) with an uncertainty of 0.2°C and a variation in successive use of less than 0.033°C. The furnace requires no inert gas or vacuum and utilizes only 225 watts to reach a melt in about an hour after turn on. Melts and freezes last about five minutes each and at least fifty melts and freezes are expected during the lifetime of the furnace.

Key words: Blackbody; optical pyrometry; radiometry.

1. Introduction

Copper-point blackbody furnaces have been made which provide sources of radiance temperature having an uncertainty of 0.2°C at 1083.3°C (Int. 1948). They were designed for use in monitoring the long term stability of the low range of automatic and visual optical pyrometers but should also have extensive use in radiometry. Emphasis has been placed on simplicity of design and construction, and the use of inexpensive materials and components.

The purpose of this note is to present such detail for the construction and assembly of such a blackbody and furnace that a minimum of time and thought is necessary to complete the work. One inexperienced in this type of work should have little difficulty. Even the required machining has been simplified so that an amateur machinist will do the work adequately. On the other hand, it is believed that the expert in furnace construction will find that a number of the techniques used will save him time in the assembly. In addition to the construction, the note presents operating procedures for the blackbody furnace and discusses its performance.

2. Construction

2.1 General Design and Description

Fig. 1 is a cross-section of the copper-point blackbody and furnace. The inside diameter of the graphite crucible is predetermined by the 1-inch diameter copper freezing-point standard reference material sample 45d, available from the National Bureau of Standards.* The length of the sample

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is 4 inches. Of its 450-gram mass, 200 is sufficient to surround the sight tube adequately; and consequently the sample is shortened. The sight tube is no longer than necessary to form an adequate blackbody cavity. This minimizes the torque on it resulting from the buoyant force of the melted copper. The cavity has an aperture or target of about 3.5 mm in diameter and the alumina cone allows a maximum exit cone of over 12 degrees (full angle).

The small projection of the ingot of copper into the back wall of the crucible lends support to the copper in its solid state. Upon cooling, the copper will contract concentrically on the sight tube and would otherwise be wholly supported by it.

The impervious alumina cone is sealed to the front of the quartz cylinder and the back of the cylinder is also sealed, thus impeding the burning of the graphite. Though air has access to the graphite through the small aperture of the cone, it has been possible to use such a furnace for over 100 hours (at least 50 freezes) before failure due to burnout.

The furnace heating element is #16 B. & S. gauge nichrome (80% nickel, 20% chromium) wire. This size wire is easy to bend, yet large enough to withstand early burnout due to corrosion. The coil has a resistance of about 5 ohms, making a medium-power variable auto-transformer adequate for supplying the required 225 watts of power to the furnace. Longer windings have been tried, but the additional heating caused the cone to expand and crack the quartz in the vicinity of the front seal. With the shortened overhang of the winding at the front of the crucible, the radiometric performance is optimized with the overhang at the back of the crucible as shown in Fig. 1.

Quartz wool is chosen for insulation within the cylinder, chiefly because of its availability in small quantities. Externally the cylinder is wrapped with 1/16 inch thick high-temperature insulating paper to an outside diameter of 4 inches. Variations of the amount of wrapping varying from 3 to 5 inches outside diameter were tried with no appreciable difference in radiometric performance. With 4 inches the outer surface is cool enough to be briefly touched by hand. The wrapping is bound with a small size (#24 B. & S. gauge) nichrome wire.

2.2 Crucible and Sight Tube

Dimensions of the crucible and sight tube are given in Fig. 2. Dimensions not shown are dependent upon fitting with the particular quartz cylinder and ceramic cone that are purchased. The crucible is machined on a lathe from a 1-1/2 inch diameter high-purity graphite rod. Use of pure graphite reduces the possibility of contamination of the copper. Since the manufacturer claims that the impurities in the graphite do not exceed 20 parts per million, care should be taken not to contaminate the graphite while working it. In particular, tools and hands should be oil free.

Inside cuts on the crucible and sight tube are made with drills. An inner diameter of the crucible of 26 mm allows for the expansion of the

1-inch (25.4 mm) copper ingot upon heating. If a 26-mm drill is not readily available, the more common 1-inch drill can be used and the resulting hole enlarged, or the copper can be machined to a smaller diameter. The outer diameter of the crucible should be 0.02 inch less than the inner diameter of the quartz cylinder to allow for the unequal expansion of the quartz and graphite. The cylinder, nominally 1-3/8 inches inner diameter, may be slightly tapered, so an average of measurements taken at both ends should be used as the inner dimension. Because the expansion coefficients of the various materials, particularly the graphite, are not sufficiently well known to achieve a close fit at 1083°C, the allowances made for both the expansion of the graphite within the quartz and the copper within the graphite are liberal.

After the outer diameter of the crucible is machined and its hole drilled, the crucible can be cut off by using a hack saw held against the turning work. This rough cut can be finished later to the specified length in Fig. 2. Meanwhile, it is convenient to fabricate the lid from the graphite remaining in the chuck. It is suggested that the central hole in the lid be drilled before the lid is machined to fit the crucible. Otherwise, strains relieved on drilling might destroy the fit desired. Graphite pieces fitted together loosen somewhat under high temperature for long periods. Therefore, the fit should be quite snug initially.

The sight tube is fabricated from a high-purity 5/8-inch diameter rod with the inner diameter drilled out before the outer surface is machined. It is advisable initially to use a drill 1/64 inch smaller than the indicated 13/32 inch for most of the cut. The lid should be tried for a snug fit onto the tube during the final cuts on the outer diameter. While the back wall of the tube is being machined to a thickness of a little over 1/32 inch, a vernier caliper indexed to 1/128 inch and a low power eye loupe are helpful in locating the start of the cut inward parallel to the drill taper. The work is illustrated in Fig. 3. The cut should start 3/128 inches beyond the inner depth. This depth can be found by measuring the length of the drill (excluding the end taper) and subtracting the protrusion of the shank of the drill when inserted in the sight tube. Parallax should be avoided in using the eye loupe. If there is uncertainty in locating the start, another 1/128 inch may be added to the outer length. The work may be cut off by using a fine-tooth hacksaw blade, and the cut finished by careful grinding or filing.

The graphite sight tube diaphragm is also made from the 5/8" graphite stock. It is drilled out to about the same size as the small end of the cone. Without removing the partly finished diaphragm from the lathe, it can be tested for a snug fit as shown in Fig. 4. The test should be made with the diaphragm at about the expected recessed position because the inner diameter of the tube may have a slight taper.

When the sight tube, diaphragm, and lid of the crucible are assembled, the diaphragm should first be inserted most of the way. To press the sight tube into the lid, it should be grasped where it is reinforced by the diaphragm. Then, with the rim of the sight tube reinforced by the lid, the diaphragm can be pushed with the cone as far as the cone will go. The cone, abutted against the graphite diaphragm and rim of the sight tube, forms a seal which reduces the circulation of air.

2.3 Copper Ingot

The copper ingot should approximate the inner configuration of the crucible to minimize voids. The end of the ingot is tapered to the 26-mm drill taper at the end of the crucible. However, considering the added difficulty, it is not worthwhile to machine the small 3/16-inch configuration to fit into the back wall (Fig. 2). The copper ingot should be shorter than the depth of the crucible by 1/16 inch to allow for the differences of expansion of the copper and graphite.

A 1/2-inch diameter hole is drilled in the ingot for the sight tube. The ingot is not likely to be straight, and in order to center the hole fairly well the ingot should be positioned in the chuck so that the drill progresses within the chuck jaws. A depth 1/32 inch more than that required to seat the sight tube provides a safe clearance between the copper and the fragile end of the tube. A sharp drill is required and a simple way to insure this is to start with a new one. Cutting oil will ease the drilling.

The ingot is reversed in the lathe and, as with the crucible, the excess length can be cut off with a hack saw preparatory to machining the back taper.

The copper should be washed clean of oil before any checks of clearances of the ingot with the crucible or sight tube are made. As a final precaution against contamination, the finished copper ingot can be immersed for a minute in nitric acid diluted to half strength, or until surface oxides are removed.

2.4 Cylinder and Cone

The quartz cylinder has a nominal inner diameter of 35 mm (1-3/8 inches) and an outer diameter of 38 mm (1-1/2 inches). However, cylinders and also cones, that are available at a reasonable cost do not have close dimensional tolerances. If the cone obtained fits loosely into the cylinder, no difficulty arises. If the cone is too large, it can be allowed to protrude from the cylinder by as much as a quarter of an inch without any detrimental effect on the furnace performance.

The specifications of the cone for this size cylinder are as follows:

Impervious alumina
Taper, 12 degrees
Small end, 1/4 inch OD
Large end, 1-3/8 inch OD
Wall thickness, less than 1/16 inch
Tolerances, average production.

Thin-walled cones of the type desired are difficult to make; therefore the thickness of the wall is not specifically given. Cones received have had apertures of from 3 to 4 mm diameter, and these were satisfactory.

A high temperature cement is required to seal the cone into the cylinder. A cement that hardens by chemical set will have the necessary strength and adherence at room temperature. The rims of both the cone and the quartz cylinder are coated with a mixture (of filler and binder) of low consistency, about that of heavy cream. The crucible is placed in the cylinder and used to position and support the small end of the cone while the seal hardens. The relative position of the crucible and cylinder should be marked so that the crucible can be replaced in the same position later. The time required for hardening may be determined by cementing together test pieces of ceramics at the same time the seal is made.

A clear quartz cylinder allows a visual check of the packing of quartz wool around the cone (crucible removed). A metal strip rounded on the end to about the inner diameter of the cylinder will be helpful in packing the insulation. The packing should be fairly firm. The space is too narrow to pack closer to the front end than about 1 inch. The small aperture of the cone should be reamed with a wet artist brush to clear out any shreds of wool.

2.5 Heater Winding

In forming the heater winding one starts with 24 feet of #16 B. & S. gauge nichrome wire. This is about two feet more than is required. The wire is straightened by anchoring it at one end and pulling it sharply such that a slight elongation is felt. A strong rod or a large screw driver can be used as a crosswise handle for this purpose.

The coil is formed on a metal tube 1-1/8 inches diameter, chucked in a lathe geared down to its lowest speed (if possible, to 48 rpm). The wire is attached to the tube by hooking into a small hole. It should be wound with no spacing, letting the wire slip through the hands with enough tension to form a tight coil on the tube. A lathe run in reverse will draw the wire over the top so that the coiling can be watched. The coil when released will spring open to a diameter somewhat less than 1-1/2 inches.

To facilitate expanding the wire onto the cylinder, a piece of graphite beveled as shown in Fig. 5 is useful. The coil is fed on, rung by rung, while the cylinder is hand rotated.

The coil, in its final position, should overhang the crucible by 3/4 inches in the front and 1-1/2 inches in the back. Variations of a quarter of an inch will not significantly affect furnace performance.

To space the rungs evenly, a soft-solder wire, 1/16 inch in diameter, is hand-wound between the nichrome rungs. The coils should be tightened by twisting with the direction of rotation. After the pliable solder wire is removed, the winding should be tightened and any rung that has moved should be repositioned.

The excess wire can now be cut off, leaving enough for the leads. After the lead wire is straightened tangentially to the cylinder and a check is made to insure the alumina protection tubing (inner bore 1/16 inch diam.) will slip on easily, the wire should be bent out along the cylinder toward the ends. After the alimina tubing is slipped on it can be fastened in place, temporarily, with some of the #24 gauge nichrome wire while each end of the tubing is spot cemented with the high temperature cement. When the cement is sufficiently dry, the windings should be brushed with a soapy solution and rinsed in preparation for complete cementing.

2.6 Completion of the Furnace

The cement used to cover the winding should be one specified for this purpose because it is more likely that it will be free of components that may unduly corrode the wire. A mixture of cement thin enough to flow in around the wires should be brushed on first; then a mixture thick enough to stay molded on the winding should be applied either with a brush or spatula. It will be helpful if the open end of the cylinder is slipped onto a large horizontal rod or wooden dowel and rotated while the cement is applied. The cement is built up about 1/8 inch from the cylinder surface and overextended on either end of the winding. An uneven surface of cement can be smoothed with a wide strip of sheet metal, lightly applied along the length of the cement while the cylinder is rotated. An occasional wetting of the strip will prevent the cement from sticking and pulling away.

Drying can be accelerated by applying 20 to 25 watts of power to the winding. When the cement is partially dry and can be cut with a knife without sticking, the ends should be trimmed back evenly leaving about 1/8 inch to overlap the winding. To insure a thorough drying of the cement, the 25 watts is then maintained overnight.

For proper insulation, end sections of the cylinder not covered with wire or cement should be covered with pieces of insulation paper to a thickness about the same as that of the cement. The seams of the pieces should abutt the alumina lead tubes. The paper can be bound in place with small nichrome wire. The entire length of the cylinder may then be wrapped with insulating paper and the furnace completed with the insertion of the crucible, packing of the back insulation, and sealing with about a 3/8-inch thickness of the chemical-setting cement.

3. Operation and Performance

A few accessories are needed in the operation of the furnace besides a variable auto-transformer. An ammeter is convenient although not essential. The maximum exit cone of the radiation is over 12 degrees and provides a considerable margin for the presently available automatic and visual pyrometers. Nevertheless, a method for aligning the pyrometer axis with the axis of the exit cone will give added confidence that the aperture stop is in the pyrometer. Crosswires have been mounted in the smaller end of a short hollow cone made of graphite, which fits into the furnace cone and centers the wires in front of the aperture. Alignment is achieved when the blackbody aperture and cross-wires are superimposed on the optic axis of the pyrometer. Although the precision of alignment is greater the farther apart the cross-wires and aperture, in some automatic pyrometers with short object distances the range of the focus may be a limitation. If cement is used to mount the cross-wires, it can be refractory cement, and alignment can be made on a hot furnace. A high-intensity microscope illuminator and a small mirror are useful in illuminating the aperture and wires for viewing. However, a flashlight is adequate.

Experience has shown that the optic axis of the furnace does not shift significantly upon heating and therefore can be aligned while cold. The edge of the diaphragm is readily visible, when the blackbody is at 1083°C, in a visual pyrometer and it can usually be seen in automatic pyrometers, though in some cases the contrast is poor. The accurate centering of the pyrometer axis in the blackbody aperture is not essential since the radiance temperature over the area of the aperture is uniform to within 0.01°C.

Fig. 6 is a typical melting and cooling curve of the copper-point blackbody furnace with power inputs of 225 watts and 110 watts respectively. At 225 watts the melt begins in about 70 minutes. When the melt has been completed the copper is allowed to overheat 5°C as determined by the pyrometer before the power is turned down. Longer melts and freezes can be obtained through appropriate power adjustments. However, the beginning and the termination of the plateaus then become less distinct.

Attempts were made to improve the curves, particularly the melting curve. On the assumption that the need for a more uniform heating of the copper along its length was indicated, the windings were lengthened or shifted, and the amount of insulation around the cylinder varied, as was mentioned previously. No significant improvement was made by variations within the restrictions of a simple design. However, the performance indicated in Fig. 6 is adequate for monitoring the best present day automatic pyrometers whose sensitivity is about 0.1°C.

For the most precise work the freezes, which have more definitive plateaus, are used. The radiance temperatures corresponding to the freezing plateau of four different copper-point blackbody furnaces are compared in the following table:

Table 1

Furnace No.	Mean radiance temp. (Int.48)	No. of freezes	Standard deviation of a freeze
1	1083.291°C	4	0.010°C
2	1083.320	19	.011
3	1083.274	9	.012
4	1083.348	5	.017
Average	1083.308		

Furnace Nos. 2 and 3 are of the construction herein described. Furnace No. 1 was made of commercially available heating elements with windings embedded in semi-cylinders. It failed after eight freezes (of which four were used with uncalibrated pyrometers) through burnout of the crucible which was evidently inadequately protected by the porous ceramic. Furnace No. 4 was constructed of a cylinder of impervious mullite and a mullite cone with the heater element extended from end to end.

Measurements were made with a photoelectric pyrometer similar to the NBS photoelectric pyrometer which has been previously described [1][1]. The pyrometer used was given a one-point calibration at 1083°C. In this calibration the sum of the absolute values of the systematic errors was 0.04°C and the standard error [2] was 0.015°C. The standard error of the pyrometer when determining a temperature is 0.006°C. Over the six month period of the work, the pyrometer was corrected for pyrometer lamp instability (discussed in ref. 1) through gold-point (1063°C) measurements on each day that measurements of the copper-point furnaces were made. The gold-point furnace was compared to an infrequently used gold-point furnace on four different occasions throughout the period with a standard deviation of the four comparisons being 0.006°C. Since the standard error of such comparisons has been determined to be 0.008°C, it was concluded that the gold-point blackbody and furnace had not deteriorated in its frequent use.

The uncertainty of the mean radiance temperature of each furnace was computed according to the recommendations of Eisenhart [2] of NBS. The standard error of the calibration process (0.015°C) was combined in quadrature with the standard error of the copper-point (0.006°C) and the gold-point (0.006°C) measurements. This value, 017°C, was tripled and added to the estimated systematic error (0.04°C) for an overall uncertainty of 0.091°C. For the purpose of predicting the uncertainty of a furnace constructed independently, variations of the design of the furnaces listed in the table are viewed as adequately bracketing any other versions of the described design including the possibility of using materials from different manufacturers. The sample standard deviation of a furnace about the average of 1083.308°C is 0.033°C*. This value, when tripled and added to the 0.091°C uncertainty, results in a rounded off uncertainty of 0.2°C.

It can be seen from the standard deviation of a freeze in Table 1 the ability to reproduce a radiance temperature with a given furnace is considerably better than the overall uncertainty. A plot of the freezes of furnace No. 2, taken over a period of five months, is shown in Fig. 7.

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[1] Figures in brackets indicate literature references in this paper.

*Any variation in the copper samples is included. However, a previous study [3] of 16 samples of the current batch of copper showed no significant evidence of nonuniformity, with a standard error for the mean of 0.011°C.

For the first 20 hours the furnace was on no more than necessary to complete the freezes and was allowed to cool down to room temperature after each freeze. Thereafter, the furnace was frequently left on for about 8 hours at a time at 1070°C or above to accelerate any deterioration or contamination. Up to 100 hours the furnace had undergone 41 freezes. All of the temperatures of the freezes are not plotted in Fig. 7; excluded are the measurements made with uncalibrated instruments. It is estimated that the deterioration of the above furnace when left on at 1070°C is equivalent to that in obtaining 10 more freezes.

The blackbody represented in Fig. 7 failed by burnout of the sight tube after 5 more freezes and at about 108 hours at high temperature. In the last 4 freezing points prior to the burnout the melting curve had shifted about 0.3°C higher than the freezing curve. The freezes had maintained their reproducibility. It might well be that a growing disagreement between the melts and freezes is a good indication the furnace should be retired.

The blackbody of furnace No. 4 gave way after 70 hours. In this case copper poured into the cone, but it solidified without dropping out of the furnace. The sight tube probably had a slight fault and was finally crushed upon cooling by the greater contraction of the copper relative to graphite.

The emissivity of the blackbody is not significantly different for variations of from 3 mm to 4 mm diameter that may be expected of the small end of the ceramic cone. A test was made in which an aperture 2 mm in diameter was, shortly after taking a freezing point, drilled out to 3.5 mm (the limits of the ceramic cone), and another freezing point taken. The measured difference was 0.022°C.

4. Summary and Conclusions

Copper-point blackbodies and furnaces of simple design have been developed and evaluated with respect to the uncertainty and reproducibility of their radiance temperature. Those constructed according to the details given may be assigned a radiance temperature of 1083.3°C (Int. 48) with an uncertainty of 0.2°C,* and a variation in successive freezes of less than 0.033°C.**

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*The quoted uncertainty refers to the radiance temperature of the blackbody and not necessarily to the freezing point of the NBS standard reference material copper. The melting range of approximately 0.1°C shown in Fig. 6 and inherent in the design precluded the possibility of reducing the uncertainty of 0.5°C reported on the certificate accompanying the standard sample copper.

**Three times the standard deviation for the variation of repeated freezes on the same furnace (based on 18 degrees of freedom).

The cost of materials purchased in the minimum allowable quantities can be expected to be between $100 and $150. There will be an excess of some materials which may be used for additional furnaces at no further expense.

5. References

[1] "The NBS Photoelectric Pyrometer and its Use in Realizing the International Practical Temperature Scale Above 1063°C", Metrologia 2, 150 (October 1966).

[2] Eisenhart, C. "Expression of Uncertainties of Final Results", Science 160, 1201 (1968).

[3] NBS Test Report on Determination and the Freezing Point of Standard Sample Copper Cu 45d, 21 November 61.

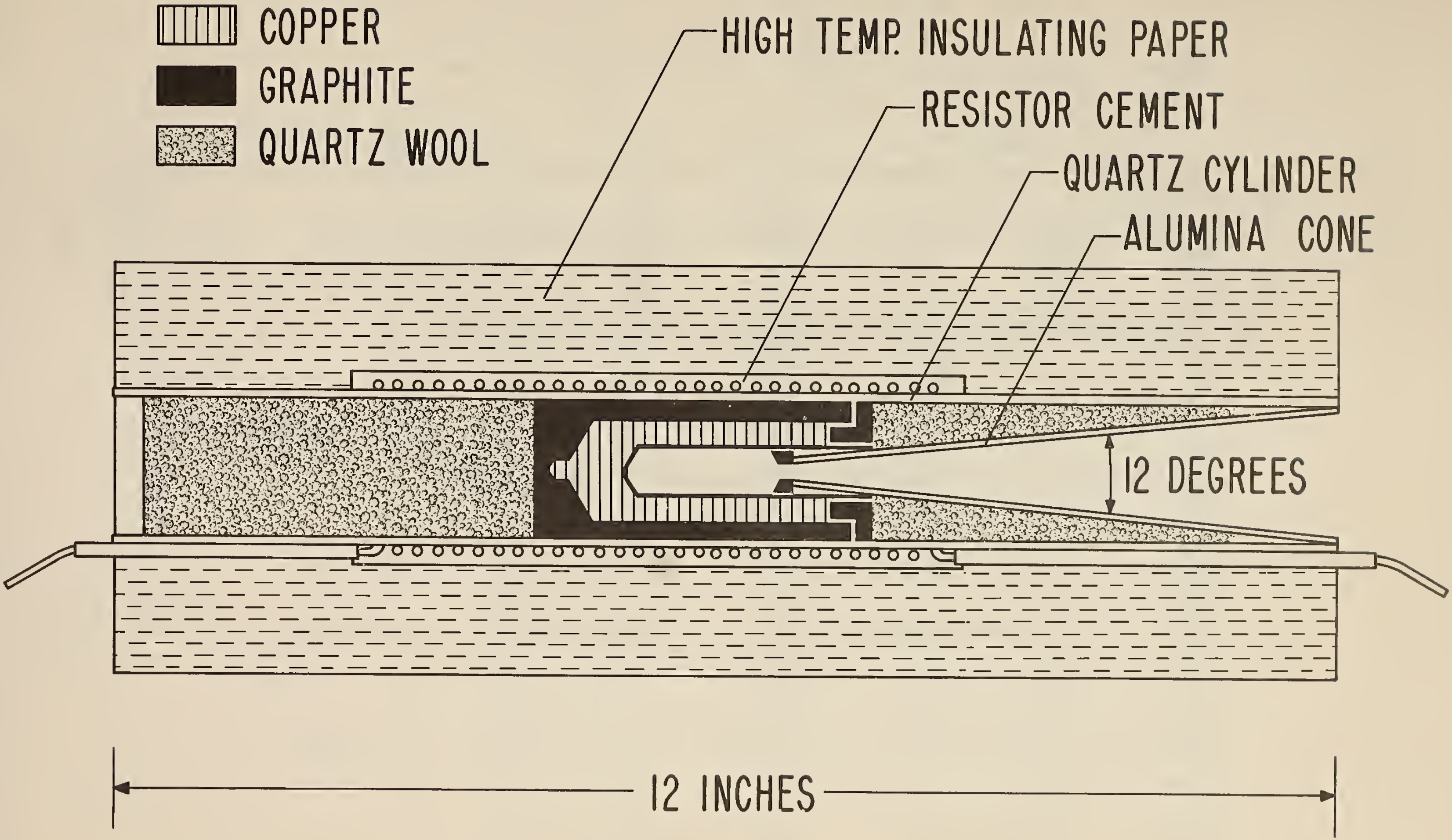

Figure 1. Cross-section of the completed blackbody furnace. The heater windings are shown enlarged for clarity.

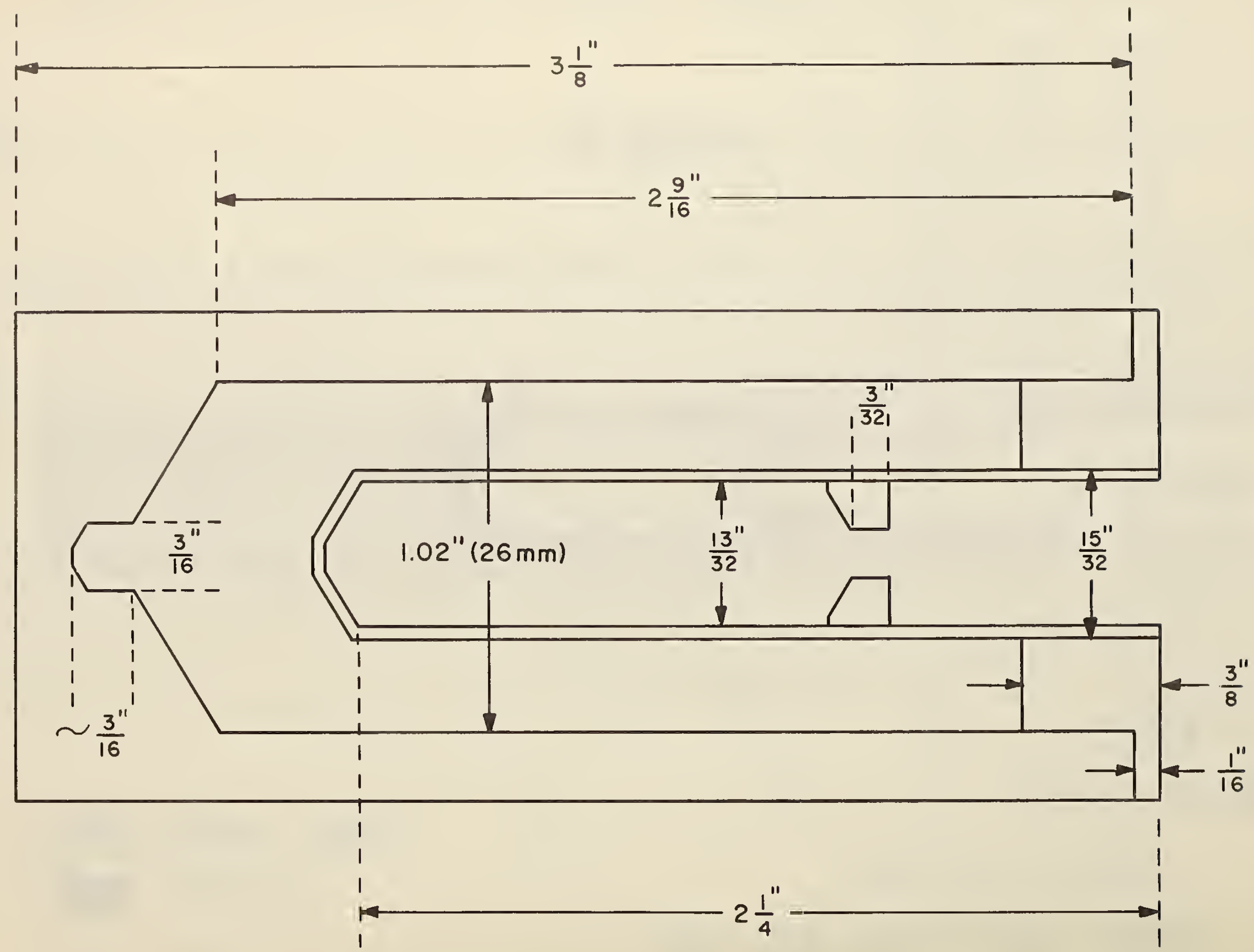

Figure 2. Cross-section of the crucible and sight tube, twice actual size. Dimensions that are not given are discussed.

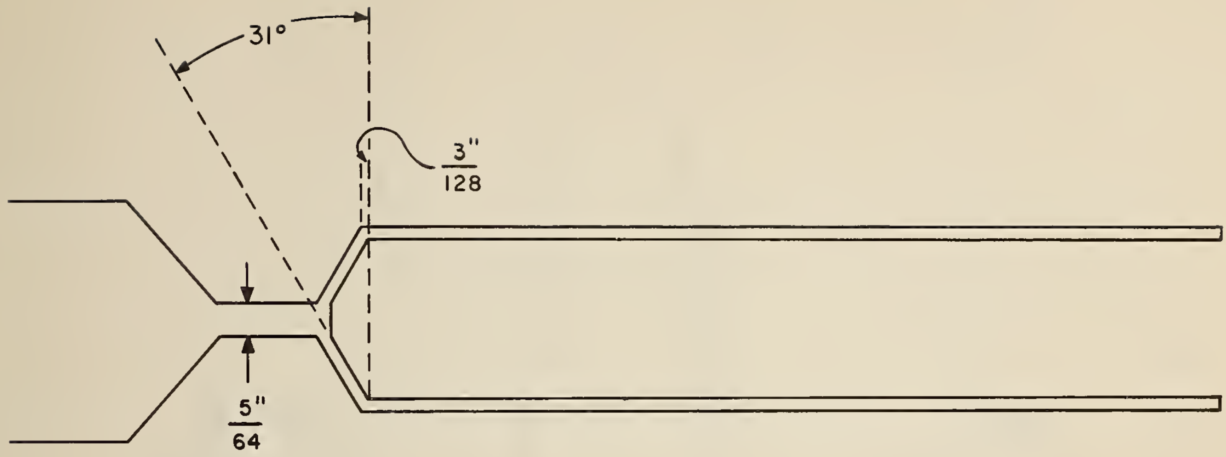

Figure 3. Sight tube ready for cut off, twice actual size. The taper is that of a conventional drill.

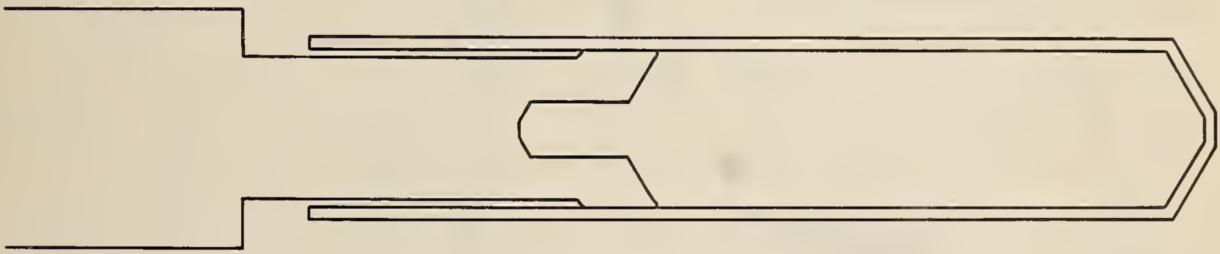

Figure 4. Checking the fit of the unfinished diaphragm in the sight tube. The diaphragm was tapered on the end with a drill. The drawing is twice actual size.

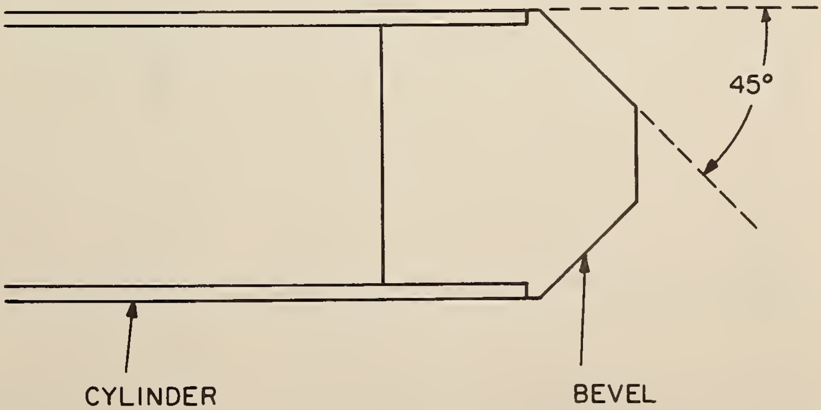

Figure 5. Bevel for expanding the nichrome coil onto the cylinder. Left-over graphite, which is easy to machine, can be used.

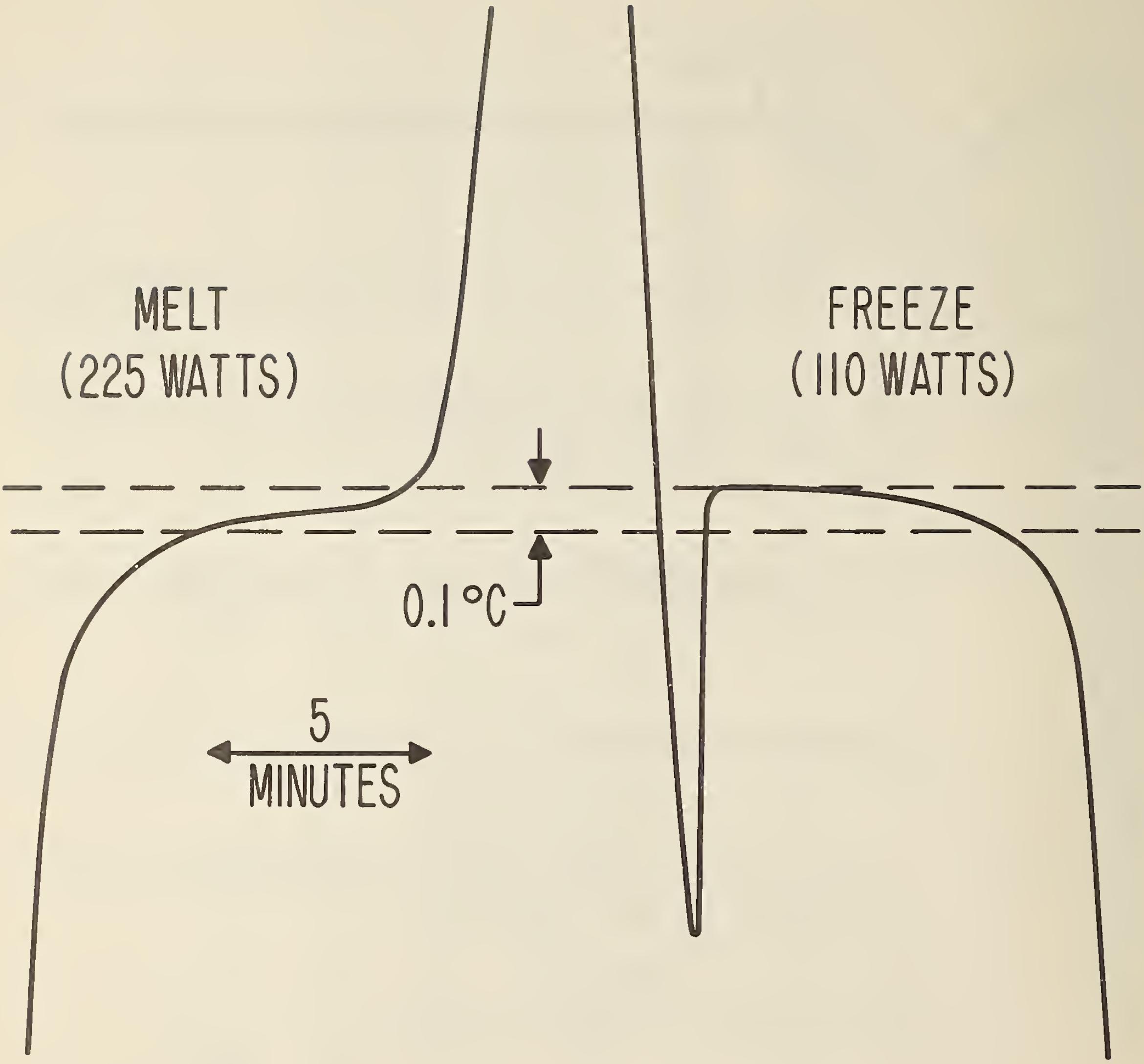

Figure 6. Heating and cooling curve of a typical copper-point blackbody.

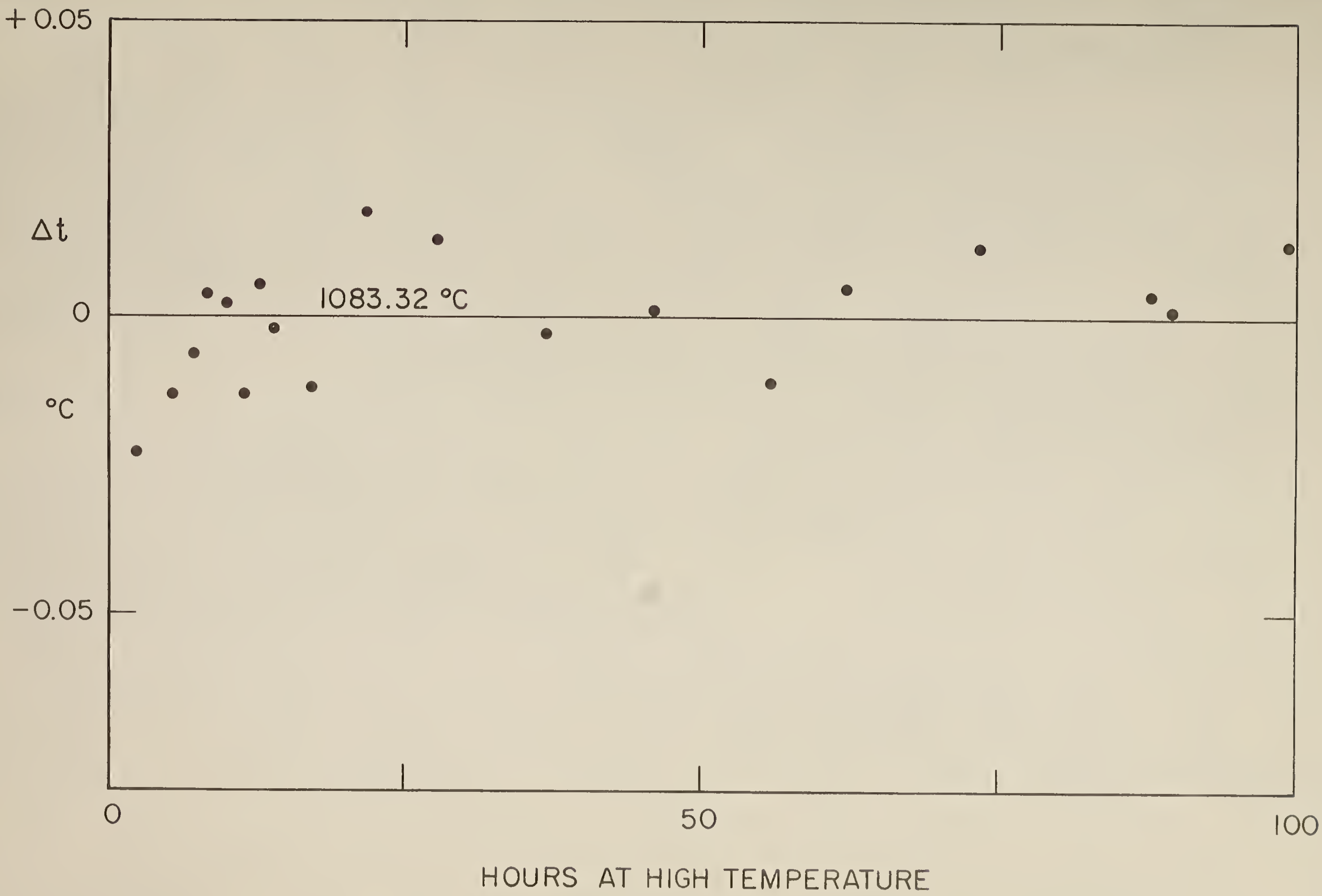

Figure 7. Freezing points of a copper-point blackbody.

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