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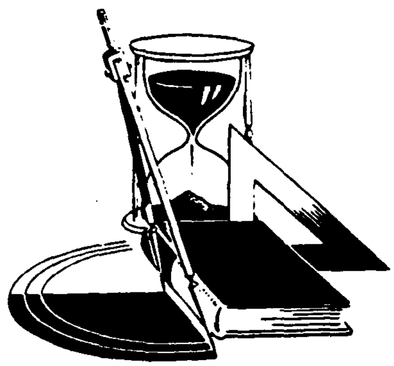

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THERMOCOUPLES . . .

Calibrating thermocouples at fixed points with tin, lead, and aluminum freezing-point furnaces modeled after those of the Bureau of Standards. Also, sulphur furnaces for immersions at 4 to 12 in.—temperature variation over this range is approx. 0.06 F

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THE increasing use of thermocouples as a temperature-measuring medium created the demand for proper calibrating facilities. As a result it was decided to obtain apparatus that would enable reliable temperature standards to be maintained. This would assure proper calibration of the thermocouples in use.

The range of calibration desired was from 32 F to 1300 F, and at about 850 F it was desired that thermocouples be checked at various immersions for use in thermocouple wells of various depths. The wells in use were from about 5 in. to 10 in. deep, so a range of 4 to 12 in. was chosen.

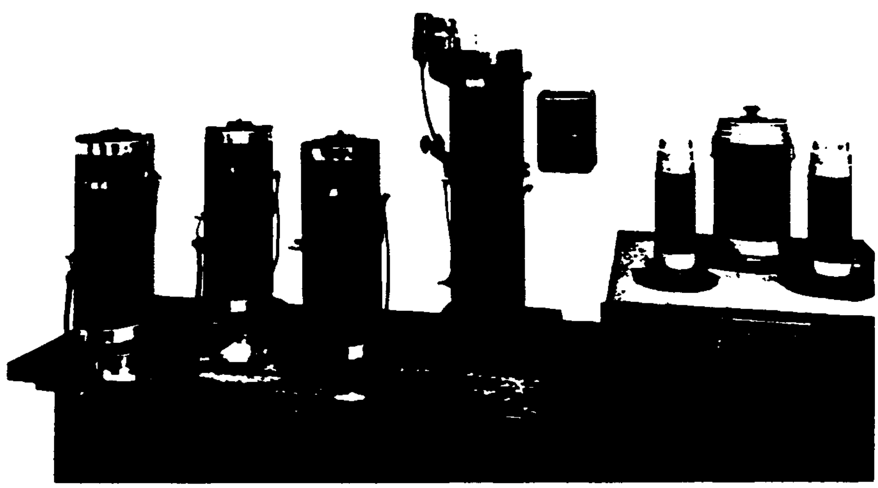

Fig. 1. Furnaces for thermocouple calibration

Within this range it seemed advisable to use the following fixed-temperature points:* The ice point (32 F), the tin freezing point (449.3$_7$ F), the lead freezing point (621.2$_3$ F), the sulphur vapor point (831 F to 834 F depending on barometric pressure), and the aluminum freezing point (1220.2$_7$ F).

The calibrating furnaces used are shown in **Fig. 1**. They consist of three freezing-point furnaces, the sulphur-vapor furnace and the ice-point vacuum bottle.

Freezing-point Furnaces

In construction, the tin, lead, and aluminum freezing-point furnaces are similar to those described in the Bureau of Standards Research paper RP768. A cross section is shown in **Fig.** 2.

A separate electric furnace of identical design is used for each metal. The heater tube is of alundum (R.A. 98) 14 in. long with an inside diameter of 2 in. and a wall thickness of ⅜ in. This tube is wound on the outside with about 70 turns of No. 14 B & S gage, Chromel "A" wire which is embedded in alundum cement. The graphite crucible containing the metal and the thermocouple protecting tube is supported by Sil-O-Cel cylinders. Two graphite diaphragms are placed above the crucible containing the freezing-of the sample and to promote better temperature uniformity within the metal bath.

The furnace shell is made of sheet steel, rolled and formed into a cylinder. The space between the heating tube and the outside shell is filled with Sil-O-Cel No. 3 insulation. The furnace top is closed with a clay graphite plate. The hot junction of a

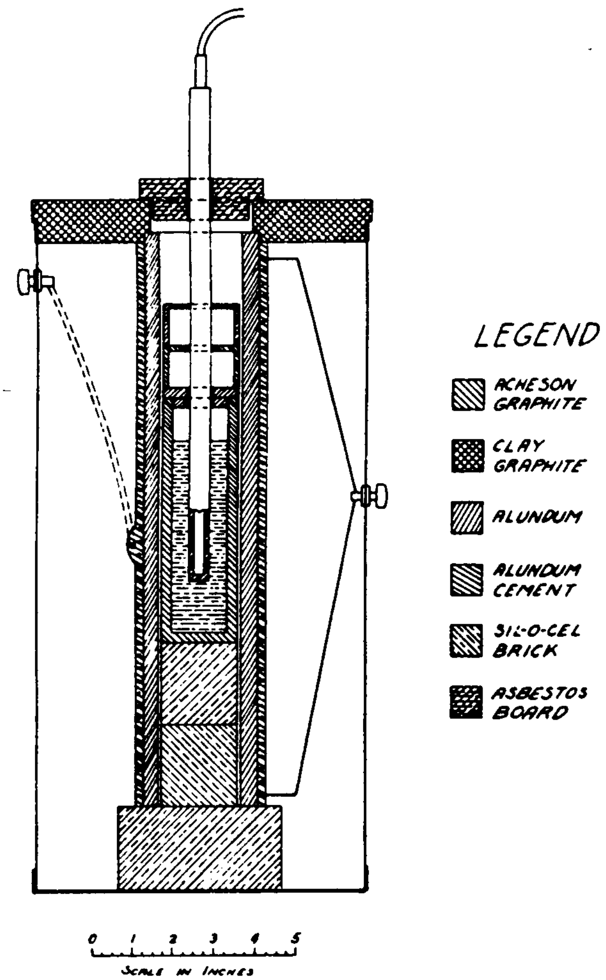

Fig. 2. Cross section of freezing-point furnace

chromel P.-alumel, No. 18 B & S gage, thermocouple is embedded in the heating element and the wires brought out to binding posts. This is used as an approximate means of controlling the temperature of the furnace to prevent overheating of the furnace winding, and to hold the metal just above the freezing point.

To calibrate the thermocouple at the metal freezing point, the thermocouple protection tube, with the thermocouple within, is passed through the furnace cover, diaphragms, crucible cover and finally rests on the freezing-point sample. The freezing-point sample is heated to about 20 F above the freezing point and held there for 15 min. After agitating the molten metal with the protection tube the furnace is shut off and the thermocouple assembly is clamped so that the hot junction of the thermocouple is about $1\frac{1}{4}$ in. from the bottom of the crucible. This leaves approximately 4 in. of the thermocouple in the metal and 6 in. more within the furnace.

Readings of the millivoltage of the thermocouple are taken, giving a time-temperature curve, which enables the freezing point of the metal to be determined. Readings of the millivoltage are taken on a five-dial precision potentiometer which is periodically checked against a laboratory-standard potentiometer with a Bureau of Standards calibration. This certifies accuracy to within 0.002 millivolts.

The metals used are certified Bureau of Standards samples. Each freezing point is double checked using a thermometer bridge and platinum-resistance thermometers. Both the thermometer bridge and resistance thermometers have Bureau of Standards certificates.

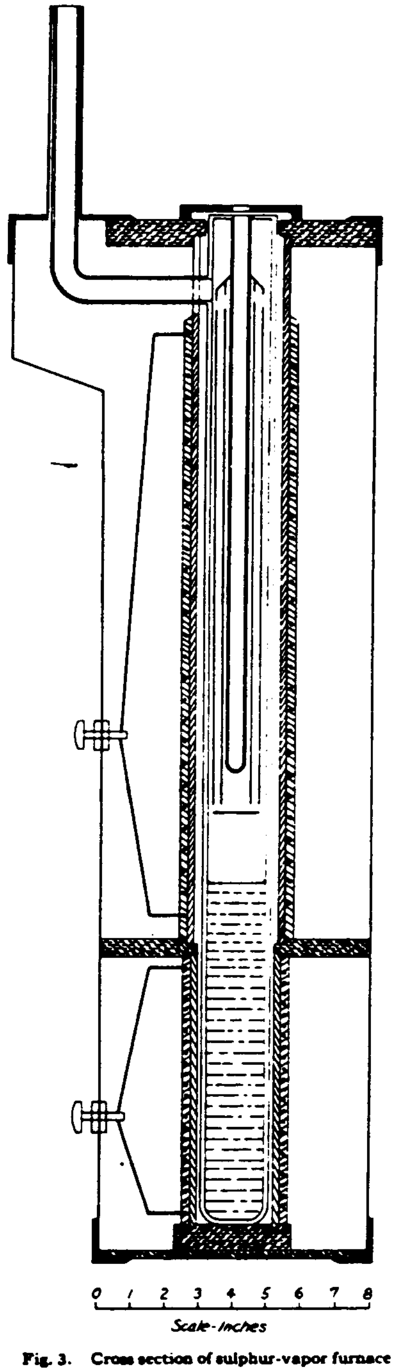

Fig. 3. Cross section of sulphur-vapor furnace

By this method the freezing points of the samples are checked within 0.04 F.

For ice point, quart vacuum bottles filled with crushed ice are used. This point is periodically checked using the thermometer bridge and platinum-resistance thermometers.

Sulphur-vapor-point Furnace

The sulphur-vapor-point furnaces, which were in use at the time this work was started, were all constructed with a single vertical tube for both the boiling and condensing of the sulphur. As a result of this construction, it was necessary to insert the thermocouple in the protection tube to same depth before it was in the region of sulphur vapor. Since it was desirable to calibrate thermocouples at immersions of 4 to 12 in., it was necessary to design the furnace to obtain these conditions. The resulting

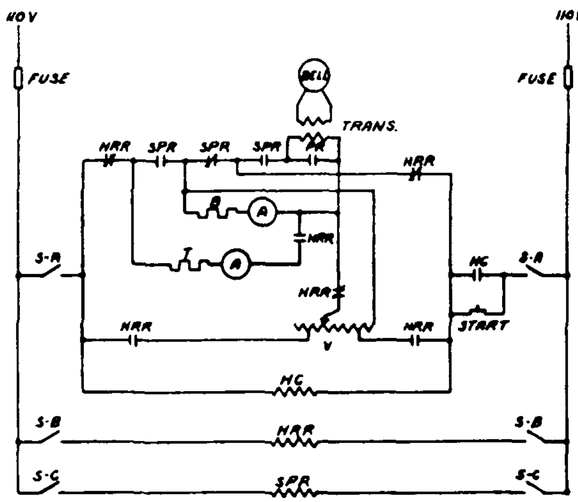

Fig. 4. Elementary wiring diagram for sulphur-vapor furnace

design is shown in **Fig. 3**, and it is seen that, by having a separate condensing tube, it is possible to obtain low immersions of the thermocouple and still be in the region of sulphur vapor.

The boiling tube is of Pyrex. It is 2 in. in outside diameter with a $\frac{3}{32}$-in. wall and is about 29 in. long. The protection tube is of Pyrex and is 16½ in. long with an inside diameter of 0.390 in. Two radiation shields of aluminum are fitted over the protection tube and are supported by a small neck in the main boiling tube. To eliminate leakage the protection tube is fused to the main boiling tube at the top, and the only vent to the air is the condensing tube which extends about 6 in. above the furnace.

The boiling tube is placed inside of a two-piece alundum core. The bottom section is 2¼-in. inside diameter by 8 in. long and carries the main heating unit, which consists of about 20 ohms of No. 21 Chromel "A" wire. The top section of this core is 2½-in. inside diameter by 21 in. long and carries a secondary unit consisting of about 30 ohms of No. 21 Chromel "A" wire. Both core sections are covered

The core and tube assembly is placed in a sheet-metal case closed at the top and bottom with asbestos board. The space surrounding the core is filled with Sil-O-Cel No. 3 insulation. To protect the top of the boiling tube, a metal plate containing a hole to admit the thermocouple is placed in the center of the top cover. A few sheets of asbestos paper are used between the boiling tube and the metal cover to reduce the thermal shock which might occur if the tube were heated more rapidly than the plate.

The condensing tube is mounted between a light source and a photoelectric relay. This is wired into the control circuit to shut off the furnace if the sulphur level rises too high in the condensing tube. This prevents the furnace from boiling over.

It has been found that sufficient U.S.P. sulphur powder must be used in the boiling tube to bring the liquid level to a depth of about 10 in. A low sulphur level will produce erratic readings due to superheating of the vapor.

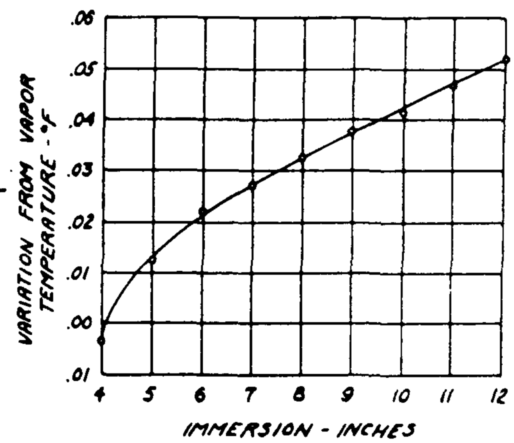

Fig. 5. Variation of indicated sulphur-vapor temperature with immersion

The control circuit is shown in the elementary diagram in **Fig. 4**. In operating this furnace certain precautions must be taken to eliminate the possibility of breaking the boiling tube. When starting, the furnace is first placed in a vertical position and turned on by closing switch *A* and pushing the start button. This places both furnace windings in series and gives a low, uniformly distributed heat which causes the sulphur to melt. After about 30 min the photocell is put into the circuit and then switch *C* is closed, putting both heating units in parallel. This raises the heat input and brings the sulphur to a boil. As the condensate level rises in the condensing tube the light beam is interrupted and the photocell relay opens, shutting down the furnace and ringing the alarm bell.

After the bell rings, switch *C* is opened and switch *B* is closed. This cuts out the secondary heating unit and puts the main heating unit across the output of the autotransformer arrangement. By adjusting the current in the main heating unit, the sulphur-condensate line is maintained about 2 in. above the on its mounting brackets so that it lays at an angle of less than 30° to the horizontal. This allows the sulphur to solidify along the side of the boiling tube.

If desired the furnace may be left overnight with switch A closed, and B and C open. This will keep the sulphur molten but not boiling. If power should fail the holding coil will open and prevent the furnace from operating again. This is necessary, since if the sulphur solidifies in the tube while it is in a vertical position, the tube may crack if it is again started. If the sulphur solidifies while the tube is in a vertical position, it is necessary to remove the tube from the furnace and melt the sulphur with a gas flame starting at the top of the tube and working to the bottom. It has been found that this will eliminate breakage of the boiling tube.

The sulphur furnace is calibrated using a four-lead potential-terminal-type platinum-resistance thermometer and an improved Mueller bridge reading to 0.0001 ohms. Both the resistance thermometer and bridge have been certified by the Bureau of Standards. For all measurements the bridge is held at 35.1 C ± 0.01 C by means of a thermostatic controller, and the resistance thermometer is checked at the ice point before and after each series of readings, the resistance being measured in both the normal and reversed positions of the bridge.

The barometric pressure is read at regular intervals throughout any test and this is used to determine the sulphur-vapor point from the formula.*

$$t_p = 832.28 + 0.16345\,(p-760) - 0.0000856\,(p-760)^2 + 0.000000079\,(p-760)^3$$

Where t_p is the temperature in degrees F and p is the barometric pressure in mm of Hg.

Results

It is assumed that the temperature calculated from the above equation is the true temperature of the sulphur-vapor point. This value is compared with the temperature determined from the resistance thermometer using Callendar's equation with the constants supplied by the Bureau of Standards. **Fig. 5** shows the results of an exploration to determine the variation in observed error with variation in immersion. This test was made in one series of runs. The only variation was in the measured resistance and a slight change in barometric pressure. It is apparent that this furnace gives a temperature reading of within 0.06 F for immersion of 4 to 12 in. Since the accuracy which is desired is only ±0.2 F the correction shown in **Fig. 5** may be neglected and the vapor-point temperature for all immersions from 4 to 12 in. can be taken directly from the given equation.

*Proceedings of the American Academy of Arts and Sciences, vol. 71, January 1937.