2. Uniform-Temperature Enclosures

Calibration of thermometers requires uniform-temperature enclosures. All of the apparatuses described here to produce such enclosures may be constructed in the laboratory, or, alternatively, may be obtained commercially. The latter tend to be more costly and to provide poorer, although adequate, uniformity. For comparison calibrations, the uncalibrated thermometer and the laboratory standard are mounted in such a way as to be in good mutual thermal contact, as in a solid metal block or a stirred liquid. Significant errors can be avoided by proper attention to immersion depths, heat transfer along thermometer leads, thermal time lags, thermometer self-heating, temperature differences within the enclosure, radiation exchange with the environment, and so on. The type of enclosure used depends upon the temperature range involved.

2.1 1 K TO 300 K

Several apparatuses that serve for the calibration of thermometers have been described. The temperature stability and uniformity of the apparatuses depend upon both their thermal design and the quality of the associated temperature controller. One that is simple, that has worked extremely well for calibrating capsule PRTs, and that can be used with germanium (Ge) or rhodium-iron (RhFe) resistance thermometers is shown in Fig. 2.1. A small copper block with holes for four thermometers is surrounded by two concentric copper radiation shields in an insulated container which is open at the bottom. The container hangs by a thin-walled metal tube in a liquid helium dewar. To prevent heat transfer along the thermometer leads, the latter pass through insulated holes in the copper block and one of the shields. Heaters are wound on both the block and the shields, and the temperature difference between them is measured with differential thermocouples. The block is cooled to liquid helium temperatures and the liquid is then blown out of the insulated container, leaving helium gas at atmospheric pressure therein. The liquid helium remaining in the dewar provides the necessary cooling. The block is heated to, and then maintained at, each desired temperature in turn, the shield temperatures being adjusted to be that of the block by a servo-control. The apparatus requires no vacuum feed-throughs for the leads and the thermometers rapidly reach equilibrium as a result of the good thermal contact between the block and the thermometer provided by the helium gas. With this system a PRT can be calibrated to within 1 mK.

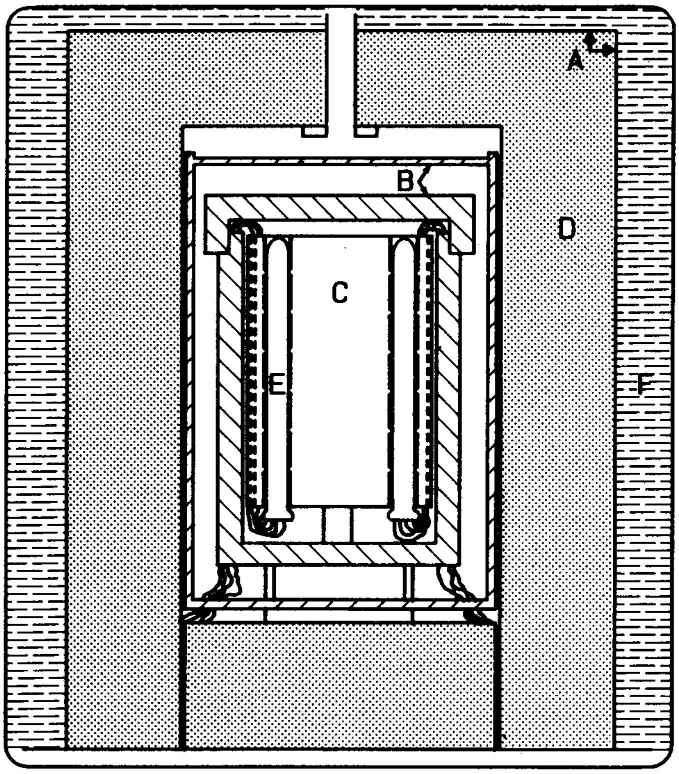

Fig. 2.1: One form of apparatus used at NRC for calibration of resistance thermometers from 4.2 K to 273.15 K: A, metal cover; B, copper shields; C, copper block; D, expanded polystyrene; E, capsule PRT; F, liquid helium.

An alternative procedure that is also widely used is to place the thermometers in a copper block that is enclosed within suitable radiation shields in an evacuated cryostat as shown in Fig. 2.2 (see, for example, Rusby et al. (1972), Compton and Ward (1975), or Besley and Kemp (1977)). Details of operation are given in the suggested references. It is sometimes useful to design the cryostat in a modular way to allow for the use of a single apparatus for different experiments. This may be facilitated by the incorporation of a cylindrical re-entrant well in the tube connecting the cryostat chamber to the part of the apparatus that is at room temperature. In this way, different modules, designed for different experiments, can be interchanged from the top flange, without one's having to disassemble the cryostat itself [see Pavese (1981)].

For temperatures above 4 K, it is also possible to use closed-cycle refrigerators, so avoiding conventional liquid refrigerants and allowing convenient and faster operation. The thermal design of measuring modules for such a system is similar to that of a flow cryostat (Fig. 2.3) [Blanke and Thomas (1979)], but care must be taken to prevent mechanical - vibrations from adversely affecting the thermometer.

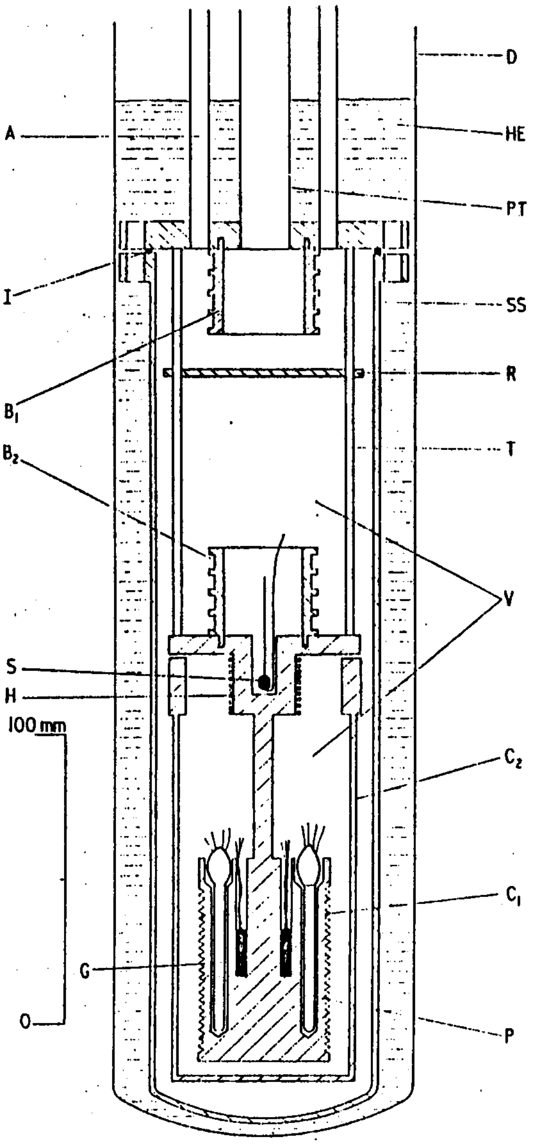

Fig. 2.2: Cryostat for calibration of resistance thermometers: D, outline of helium dewar; HE, liquid helium reservoir; PT, pumping tube; SS, stainless-steel vacuum can; I, indium seal; R, radiation shield; A, one of four access tubes for wires; B, binding post for wires; T, one of four support tubes; V, vacuum; S, temperature control sensor; H, heater; C 1, copper thermometer block; C2, copper isothermal shield; P, platinum thermometer; G, germanium thermometer [after Rusby et al. (1972)].

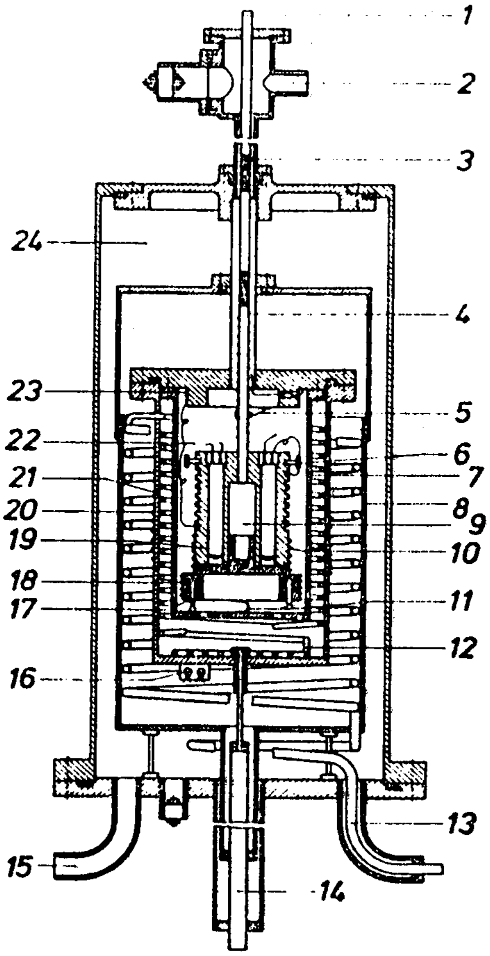

Fig. 2.3: Flow cryostat, shown with a vapour pressure bulb fitted: 1, pressure tube with radiation traps; 9, bulb with thermometers; 15, 24, vacuum space pumped through; 8, outer shield, nitrogen cooled through inlet 13 and coil 7; 20, inner shield, helium cooled through syphon 14 and coils 12 and 21; 23, isothermal shield, with internal independent vacuum 22; 16, thermometers : for temperature regulation of the shield. [Blanke and Thomas (1979)].

The sealed-cell cryostat of Ancsin and Phillips (1984) can also be used as a comparator over the temperature range 84 K to 400 °C (see Sec. 3.1.4).

In any of these systems, it is important that the thermometers be in good thermal contact with the block, as through the use of a vacuum grease. It is also important to prevent heat exchange between the thermometer and room temperature via the connecting leads; for this reason the thermometer leads, *and* an appropriate length [Hust (1970); Fellmuth and Seifert (1988)] of the connecting wires must be put in good thermal contact with the measuring block or with a body maintained at block temperature. However, this procedure must not prejudice the maintaining of a sufficiently high electrical insulation resistance. The necessary resistance depends upon the accuracy required and the thermometer resistance. In measuring the insulation resistance, care must be taken not to damage the thermometer by too-high currents from the high-resistance-measuring device. In general, the minimum allowable insulation resistance is tens of megohms. The wires coming into the experimental chamber of the cryostat should also be thermally anchored at the refrigerant temperature. A suitable thermal-anchoring material is made of copper evaporated over a thin plastic foil such as Kapton.

2.2 150 K to 350 °C

In this range the most convenient enclosures for mounted thermometers are stirred-liquid baths. The approximate temperature ranges that may be covered by various liquids are: 150 K to 225 K, isopentane; 225 K to 20 °C, methyl alcohol; 2 °C to 90 °C, water, with the range extendable to -5 °C by the addition of a small quantity of glycol; 50 °C to 350 °C, mineral or synthetic oils. The essential features of a bath used at NRC are shown in Fig. 2.4. The liquid is contained between 50 cm long concentric cylinders of diameters about 10 cm and 6 cm respectively. A propeller driven through a seal at the bottom of the bath and with appropriate anti-spin vanes forces the liquid up the inner cylinder and down between the two. An immersion heater between the cylinders controls the temperature and a second heater on the outer cylinder maintains the external temperature a few degrees below the required calibration temperature. For the water bath, cooling is obtained by circulating Freon through the refrigerant coils shown on the outer shell. With the oil bath, which operates only above ambient temperature, the refrigerant is unnecessary. For the alcohol or isopentane baths, liquid nitrogen or solid carbon dioxide are the usual refrigerants. The thermometers to be calibrated are mounted at suitable depths within the inner cylinder. Uniformity of temperature to better than 10 mK, and with care to within 1 to 3 mK, within this inner cylinder is readily achieved. Other variations of this general design are described by Quinn (1983) and Pavese and Coggiola (1972).

With the oil baths, light mineral oils are used up to about 200 °C, and heavy oils up to about 350 °C or even 375 °C on occasion. The upper limit is set by the flash point of the oil or by the tendency of the oils to carbonize. The lower limit is that at which the viscosity has become too high for efficient heat transfer to be maintained. In using an oil bath, it is essential to provide a proper venting system to remove the noxious fumes from the laboratory. It is also advisable to keep a suitable fire extinguisher at hand if the bath

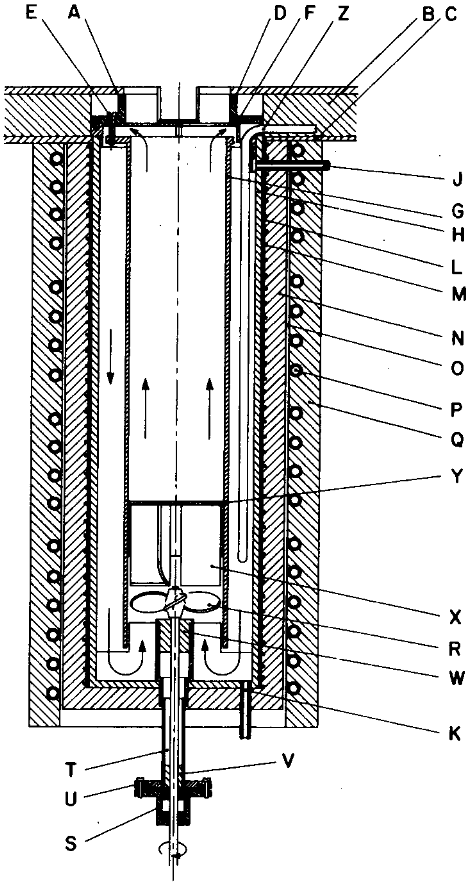

Fig. 2.4: Stirred liquid bath for use from -5 °C to 350 °C: A, top cover, bakelite; B, insulation, expanded polystyrene; C, bakelite; D, top ring, bakelite; E, weir height adjustment screw; F, splash shield; G, weir, brass; H, water container, brass; J, overflow tube, german silver; K, drain, german silver; L, insulation, mica; M, outer heater; N, insulation, fiberglass; O, copper sheet; P, refrigeration coil; Q, insulation, fiberglass; R, propeller, 4 blades brass; S, closure housing including seals and lubricants; T, tube, german silver; U, o-ring seat; V, lower bearing, teflon; W, upper bearing, teflon; X, anti-spin vane; Y, screen, copper.

temperature is to be raised to near the flash point because ignition may occasionally occur below the expected flash point.

2.3 300 °C to 1100 °C

For temperatures above the range of stirred oil, no completely satisfactory bath is available. A salt bath (containing a mixture of equal parts of potassium nitrate and sodium nitrite) will provide an extremely uniform temperature from about 250 °C to 550 °C, but has substantial drawbacks. These include: a highly corrosive action on the bath container and on any devices inserted into the salt (a quartz-sheathed thermometer must be protected from the salt by an auxiliary Pyrex protection tube); a salt residue left on a device after calibration which is often difficult to remove; a potentially dangerous condition during heat-up if expansion of liquid is prevented by entrapment in solid salt; an extremely long heat-up time for the solid phase; possible violent splatter when foreign (organic) materials come in contact with the salt; requirement for venting fumes; and a generally messy housekeeping problem. Industrial safety codes for the operation of salt baths should be consulted [for example, HMSO (1964)].

An alternative to the salt bath is a fluidized bed, or fluidized powder bath. This can be capable of operation over an extremely wide range (from below ambient to 1100 °C) but it is difficult to obtain within the working volume a temperature that is uniform enough. The fluidizing medium is usually very finely powdered aluminium oxide through which dry air, nitrogen, or argon is allowed to flow slowly upward from an underlying porous diffuser of metal or ceramic. When fluidization begins, the individual Al_2O_3 particles circulate freely, and the powder takes on many of the properties of a liquid. The appearance is much like that of swirling cream with gentle surface bubbling. The powder tends to rise and migrate slowly from the centre to the walls. Heat is supplied in much the same way as in a liquid bath. Frequently, in the higher temperature units, a layer of zirconium oxide that does not fluidize but assists in the diffusion and inhibits the burning of the porous plate lies between the Al_2O_3 and the solid diffuser plates. Typical temperature differences in the fluidized bath range from tenths of kelvins to as high as a few kelvins. If a metal block is used within the fluidizing medium, better uniformity is possible. In addition to a frequently inadequate temperature uniformity, another disadvantage is the deposition of Al_2O_3 dust on everything near the bath. Care must be taken to avoid mechanical vibrations arising from the circulation of the fluid powders.

Wire-wound resistively-heated electric furnaces are the most common uniform-temperature enclosure above the range of liquid baths. These are essentially the same as those described in "Supplementary Information for the ITS-90" [CCT (1990)] for the realization of metal freezing points. A metal block (aluminium, copper, Inconel) is contained within a cylindrical ceramic muffle on which the heaters are wound. The main heater runs the whole length of the muffle, while smaller ones are placed either over it or within the muffle near each end, compensating for heat losses and thereby lengthening the uniform-temperature zone. The whole is suitably insulated. With care, for a 60 to 90 cm furnace, it is possible to adjust the heater powers so as to obtain a central 20 to 30 cm zone that is uniform in temperature to some tenths of a kelvin. The convenient upper temperature limit for such furnaces is 1100 °C, up to which Nichrome or Kanthal heater windings have long lifetimes. For higher temperatures one must resort either to expensive platinum-alloy heaters or to refractory-metal heaters that require protection from air or oxygen.

A simpler arrangement with even better temperature uniformity uses a cylindrical heat-pipe liner within the furnace muffle. In this case a single heater will suffice. A heat pipe is essentially a closed tube or chamber whose inner surfaces are lined with a porous-capillary wick. The wick is saturated with the liquid phase of a working fluid and the remaining volume of the tube contains the vapour phase. Applied heat vaporizes the working fluid in some sections, resulting in a pressure difference that drives vapour to cooler regions where it will condense, releasing the heat of vaporization in that section of the pipe. Depletion of liquid by evaporation causes the liquid-vapour interface to enter into the wick surface. The capillary pressure developed there pumps the condensed liquid back for re-evaporation. That is, the heat pipe can continuously transport the heat of vaporization from the evaporator to the condenser section.

The amount of heat that can be transported in this way is several orders of magnitude larger than that which can be transported in a conventional system; the heat pipe acts as a tube of extremely high thermal conductivity. Hence, when it is placed within an already-heated furnace it increases the uniformity immensely. Quinn (1983) gives a useful, more detailed discussion. The working fluid is usually sodium or potassium for heat pipes of interest here. A metal block may be placed within the heat pipe, but in many cases may be unnecessary. Care must be taken in the use of metal-enclosed sodium or potassium in heat pipes; the usual precautions for use of these metals should be taken.

A more sophisticated use of the heat pipe is in the pressure-controlled or gas-controlled heat pipe [see, for example, Bassani et al. (1980)]. The cylindrical heat pipe is installed in a conventional electric furnace as above, but in this case most of the applied heat is concentrated near one end (the concentrator); a vertical (or tilted, for vertical furnace operation) water-cooled chimney (the condenser) is attached at the other end; and an inert buffer gas (helium) is maintained over the working fluid (sodium) vapour, the buffer-gas pressure being externally controlled by conventional techniques. The furnace temperature can be changed from one level to another by changing the buffer-gas pressure; re-equilibration at the new level is rapid, typically within a few tenths of a kelvin in 20 minutes for a 50 K change. A standard pressure and temperature feedback control system allows the temperature to be controlled at a given level to within a few mK. The response to small pressure changes is very rapid, the speed being determined more by the temperature-sensor response than the heat-pipe response. Temperature uniformity within the furnace to within a few mK is possible. With sodium as the working fluid, the heat-pipe furnace is operable from about 550 °C to 1100 °C; with potassium, from about 400 °C to 950 °C.

Another type of furnace used for the calibration of thermocouples is the electric tube furnace, shown schematically in Fig. 2.5 [Dauphinee (1955)]. The cylindrical Nichrome furnace tube about 3 cm in diameter and 50 cm long forms part of a one-turn secondary of a high-power transformer, and is resistively heated by the high ac secondary current. A silica tube is placed inside the furnace tube, and the thermocouples to be calibrated are inserted inside this with their tips welded or otherwise physically joined together. The furnace is uninsulated so its temperature can be changed very rapidly. A typical calibration run with Pt10Rh/Pt thermocouples takes 2 hours from ambient to 1100 °C and down again. This high speed is possible because the absolute emfs of the unknown and the standard need not be simultaneously known, but only the slowly-changing difference between them. Typically, with Pt10Rh/Pt calibrations for example, this difference when the furnace is heating will have slightly different values from when it is cooling, but it can be shown that the mean of the two is the same as the steady-state difference. With (even slightly) dissimilar base-metal thermocouples, the furnace temperature must be stabilized at the desired calibration points before the emfs are measured. In this furnace there is really no constant temperature zone, but this does not matter so long as the small volume around the welded tips is isothermal.

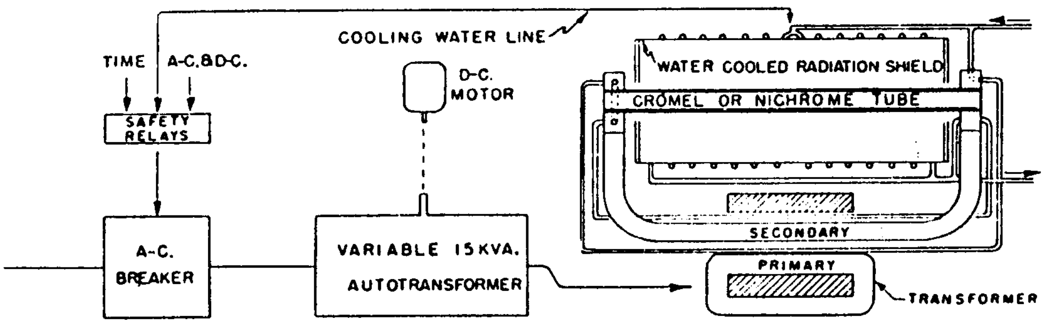

Fig. 2.5: Schematic drawing of a fast-response electric tube furnace for thermocouple calibrations [Dauphinee (1955)].