Measurements of the temperature distribution of liquid nitrogen contained in 15 and 7.5 cm diameter dewars show that the bulk of the liquid is at an approximately constant temperature .15 to .3 K above the surface temperature. About half the liquid in the dewar is in a superheated state and this state, allthough easily disturbed from equilibrium by thermal and mechanical perturbations, occurs in an unclean vessel and with liquid nitrogen likely to contain impurities. The usefulness of this phenomenon is demonstrated by showing that an immersed copper waveguide was within a volume (50 cm long by 15 cm diameter) of liquid nitrogen that was isothermal to ± 10 mK.

The temperature of liquid nitrogen in cryostat dewars

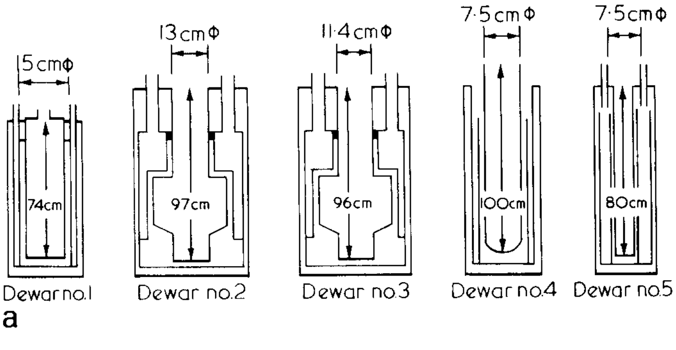
D.J. Blundell and B.W. Ricketson

A microwave thermal noise source, consisting of a resistive termination in a rectangular copper waveguide immersed in liquid nitrogen, has been described by Blundell.[1] If such a blackbody is used as a standard, it is necessary to know the absolute temperature to .05 K if an accurate calculation of the output-power spectral density is to be obtained. This paper describes measurements made on the temperature distribution in various cryostat dewars with annular nitrogen shielding to establish the thermal conditions in which a microwave source would operate.

The investigation was carried out in different laboratories by the two authors using different measuring equipment.

Dewars and measuring equipment

Five dewars were used in the investigation, three dewars had a nominal 15 cm internal diameter and one, 7.5 cm internal diameter. Their construction, dimensions and nomenclature are indicated in Fig. 1. All the dewars had an annulus for liquid nitrogen shielding. Gas tight top plates had provision for the insertion of temperature probes and the collection of boil-off gas for evaporation rate measurement. Notice the enlarged section of the inner cans of dewars No. 2 and 3, which originally housed helium-cooled masers.



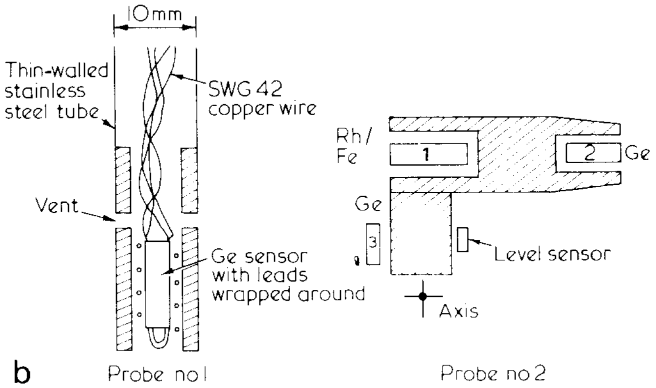


Fig. 1 Experimental apparatus. Approximate dimensions and nomenclature of the five dewars investigated with probe layouts

Two types of probe were used. The simpler had a single CryoCal Inc. type CR2500H germanium sensor fixed on the end of a thin walled stainless steel tube. The other had three sensors mounted on a support which allowed rotation in a horizontal plane (Fig. 1). The latter probe carried two CR2500H sensors and a miniature Rhodium-Iron sensor. The electrical connections were made with 0.1 mm enamelled copper wire. The sensor leads were shortened and in the simpler probe were wrapped around the sensor. In the other probe, the leads were led for at least 50 mm along the horizontal support. Above and below the liquid surface a steep temperature gradient exists (10 and 50 mK cm^{-1} respectively). The simpler probe did not measure temperatures near the surface satisfactorily, but the second measured about 20 mK high, as compared with the temperature calculated from the atmospheric pressure (T_{FS}). This difference was considered satisfactory as showing little heat input via the electrical connections, particularly as there was no shield from room temperature radiation.

The sensors were calibrated to an accuracy of ± 8 mK of the IPTS (68) scale between 65 and 100 K.[2] The germanium sensor calibration data for both ac and dc excitation was fitted to the formula $R = A + BT^{-m} + CT^{n}$ to better than 1.5 mK rms deviation.[3] Of the six germanium and the two Rhodium-Iron sensors used, all remained stable except for three

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D.J. Blundell is with the Royal Signals and Radar Establishment, Malvern, Worcs, UK and B.W. Ricketson is with Cryogenic Calibrations Ltd, Pitchcott, Aylesbury, Bucks, UK. Received 28 August 1978.

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germanium sensors, which on recalibration showed shifts of approximately 40, 30 and 5 mK.

To measure the sensors' resistance, one author (DJB) used a dc constant current source, a highly linear dc amplifier with a gain of 1000 and a 6½ digit DVM. A minicomputer was used to calculate and print sensor temperatures. The amplifier noise limited resolution to about 3 mK for the germanium sensor and 7 mK for the Rhodium-Iron with a current of 500 μA flowing through all the sensors. Experience showed that the best repeatability was obtained by averaging over five scans taken at 1 minute intervals. The other author used a self-balancing 25 Hz potentiometer (Cryobridge) with an instantaneous printout of the voltage across the sensor and the standard resistance. With a current of approximately 300 μA, 1 mK could be resolved on all sensors. Results with both the ac and dc methods of measurement agreed well. It was important to move the sensor probe as little as possible and only upwards, if disturbance to the temperature distribution was to be avoided. At least two to three hours (and often overnight) were allowed before measuring to ensure equilibrium.

No particular attention was paid to the cleanliness of the dewars or the extent of contamination of the liquid nitrogen.

Preliminary experiments

It was originally supposed that the temperature in liquid nitrogen would rise with increasing depth beneath the surface such that vapour-liquid equilibrium was maintained. From density and vapour pressure data, this temperature gradient can be shown to be 6.1 mK cm^{-1}. Measurements in No. 5 dewar with sufficient helium gas in the vacuum space to produce approximately 25 W heat input showed this to be true. Measurements in a glass dewar also indicated a slope of 6 mK cm^{-1} but about 0.1 K high.

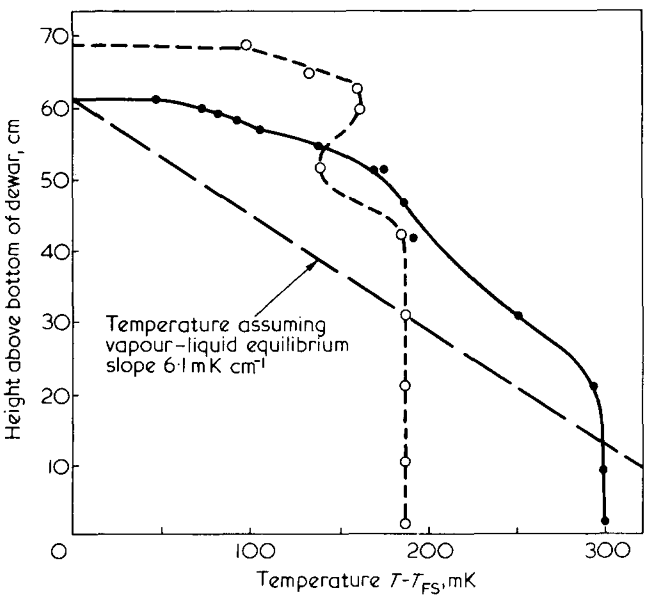

Fig. 2 Axial temperature distribution in No. 5 dewar. Solid line — without liquid nitrogen shielding. Total heat input 4 W, temperature fluctuations approximately 5 mK. Broken line — with liquid nitrogen shielding. Total heat input, 1 W, temperature fluctuations less than 1 mK

Fig. 2 shows the axial temperature distribution in No. 5 dewar. The solid curve was measured two hours after filling the can of the dewar and shows a steep temperature rise close to the surface, followed by an approximate rise of 6 mK cm^{-1} and an almost isothermal region in the bottom 20 cm of the dewar. The heat input was 4 W. Liquid nitrogen was poured into the annulus and the dewar can topped up. Half an hour later the temperature up to within 3 cm of the surface, was constant to within ± .05 K at a mean $T - T_{FS}$ of 0.25 K. Three hours later the mean temperature had appeared to come to equilibrium at a lower temperature, the axial distribution being shown as the broken curve in Fig. 2. The heat input was about 1 W.

From the preliminary experiments, it can be seen that as the heat input into the wall of the inner can is reduced, the temperature distribution tends from that expected for a vapour-liquid equilibrium system to an isothermal condition.

The temperature in 15 cm diameter dewars without a waveguide

The temperature at any given position within the dewar was not usually steady and the variations in temperature were not reproducible, either between experiments or dewars. The temperature changes could be divided into short, medium and long term.

Short-term. With 25 and .25 Hz bandwidth, rms deviations of about 4 and 2 mK were obtained. These figures would be dependent on the thermal inertia of the sensor. Temperature fluctuations were much higher towards the surface.

Medium-term. Fig. 3a and b illustrate two types of temperature variation with cycle times of between four and seven minutes. This variation is almost certainly associated with

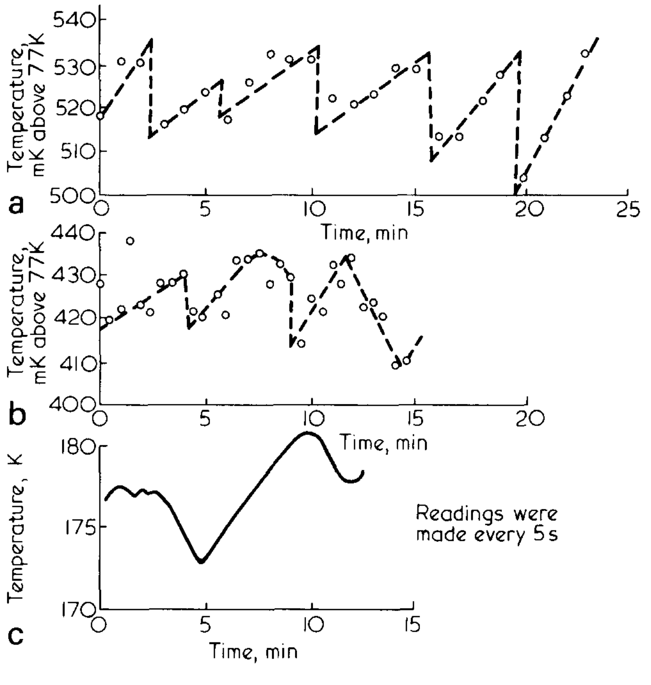

Fig. 3 Medium-term temperature fluctuations. Graphs a and b show variations in the liquid with cycles times of 4 to 6 min. Graph c shows the variation of a sensor approximately 10 cm above the surface of the liquid in No. 4 dewar. Heat input — 2 W

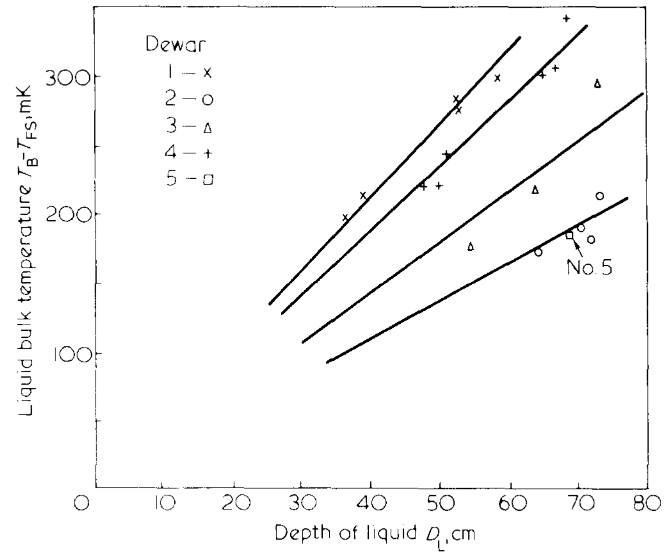

Fig. 4 Variation of bulk temperature (T_B) with liquid depth (D_L) for all the dewars. Heat input always dropped with level, but there is no simple correlation between $T_B - T_{FS}$ and heat input of the various dewars

an irregular boiling rate as is indicated by the similar variation in the boil-off gas temperature (Fig. 3c).

Long-term. Variations in atmospheric pressure would have altered the surface temperature by over 0.1 K. Results are presented relative to this calculated surface temperature (T_{FS}).

The variation of the bulk mean temperature (T_B) for various depths of liquid (D_L) is shown in Fig. 4 for dewars No. 1 to 4. There is no correlation between the measured temperature and the heat input as calculated from the evaporation rate. The results in Fig. 4 can be summarized by the equation

$$T_B - T_{FS} = KD_L$$

where K remains constant during a particular experiment.

The variation of mean temperature with heat input was measured for dewar No. 4 by allowing the pressure to rise in the vacuum space (Fig. 5). For the highest heat inputs, the temperature fluctuations were ± .05 K but there was no switch back to a vapour-liquid equilibrium state.

Outside the medium-term temperature fluctuations (approximately ± 40 mK), no variation in the radial distribution could be measured. Due to the size of the sensors, any boundary layer less than 5 mm thick would not have been detected.

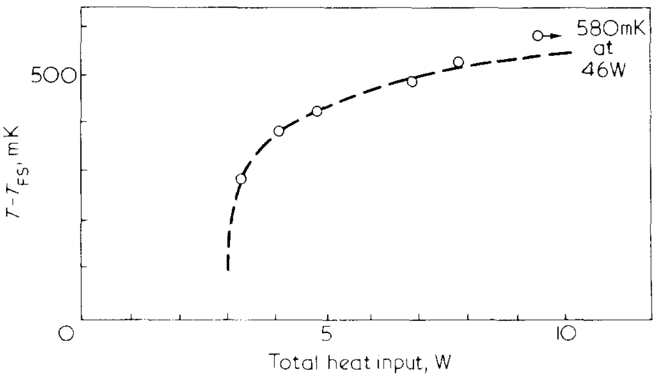

Fig. 5 Variation of the bulk temperature of the liquid nitrogen with heat input. Nitrogen gas was allowed to leak into the vacuum space. No. 4 dewar

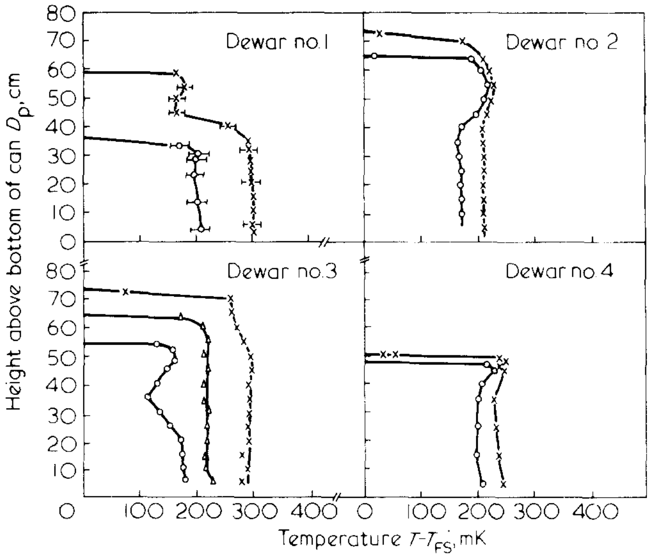

Fig. 6 The axial temperature distribution in dewars No. 1 to 4 with liquid nitrogen shielding. Only some of the curves are shown for each vessel. The ±1-sigma spread in temperature readings is shown with the results of dewar No. 1 and is representative of the spread for all the dewars

The axial temperature distribution of dewars 1 to 4, all with annular liquid nitrogen shielding is given in Fig. 6. Measurements taken ten weeks later gave slightly different bulk temperatures (not shown), perhaps because of a change in the quality of the liquid nitrogen. This has not been confirmed.

Temperature in dewar no. 4 with waveguide

A copper waveguide, 63 cm long and 4.69 cm^2 in cross-section was suspended axially on a stainless steel waveguide from the top plate of No. 4 dewar. Temperature measurements were made on probes inside and outside the waveguide and in the bottom flange of the waveguide.

There were no short or medium term fluctuations, measurement to within 1 mK being possible except within about 5 cm of the surface. There were long fluctuations of 20 to 40 mK which could be associated with the 'cold' liquid nitrogen

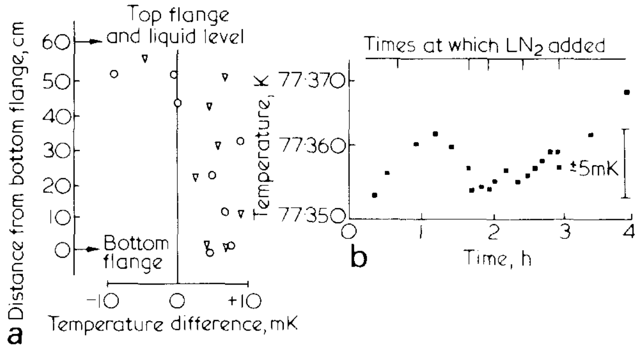

Fig. 7 Waveguide in No. 4 dewar. a — Temperature difference between sensor in bottom flange of waveguide and probe at various positions in dewar. ⊙ — with probe rising, ▽ — with probe falling. b — The temperature of the bottom flange over four hours showing times when liquid nitrogen was added ('topping up')

used to top up the dewar. Within these bulk fluctuations, the bottom 50 cm of the waveguide was in an isothermal volume to within ±10 mK as shown in Fig. 7a where the differences in temperature between the outer probe and flange thermometer are plotted. The main disturbance to the temperature being the 'topping up' procedure, a splash cup was installed to stop the added liquid nitrogen from falling directly onto the surface. By adding small quantities of liquid at short intervals it was found possible to keep the temperature of the flange to within ± 5 mK over three hours (Fig. 7b).

Discussion

The existence of quasi-isothermal conditions in dewars of liquid nitrogen or any other boiling liquid, cryogenic or otherwise does not seem to have been reported. (A search through Dialtech computer literature sources led to no relevant references.) The small temperature differences and the requirement of accurate absolute calibration of the thermometers has perhaps militated against identification of the phenomenon. There are two aspects requiring explanation; firstly, the presence of an apparently stable volume of superheated liquid at the top of the dewar and secondly, the subcooled liquid in the bottom half of the dewar.

Very pure liquids can be superheated if there are no suitable sites for bubble growth. Liquid oxygen, which has been made very pure for many years, will usually superheat in a glass dewar perhaps 1 or 2 K before releasing its excess enthalpy by rapid boiling. This boiling can sometimes occur violently. Liquid nitrogen does not appear to behave in this manner.

If superheating occurs, it can be considered a stable condition if there is no net quantity of heat flow into the liquid to raise the temperature to that which will trigger bubble growth. Without a liquid-gas phase change, the dominant heat transfer mechanism will be convection.

Heat transfer into a dewar with liquid nitrogen shielding is almost totally by radiation from the top plate and conduction down the wall of the can. Liquid nitrogen is transparent to infra-red radiation so heating of the main bulk of the liquid is only via the absorbed radiation on the wall. Heat conducted down the wall (it is probably around 0.1 W in these experiments) is absorbed in the first few centimetres below the surface of the liquid. This gives rise to a steep temperature gradient with height, say ΔT, that will allow the heat to be transferred to the surface. Very close to the surface, heat is released by boiling. The bubbles are very small and disturb the surface so little that the bottom of the dewar is clearly visible. If we assume that there is no flow into the bulk of the liquid, the liquid must eventually take up the temperature $T_{\mathrm{FS}} + \Delta T$, where T_{FS} is the temperature at the surface of the liquid. Any liquid hotter than its surroundings will rise (convection) and either mix with colder liquid or release its heat at the surface at the temperature $T_{\mathrm{FS}} + \Delta T$. The convection process can only carry to the surface a given quantity of heat. If the heat flow into the liquid is greater, the temperature will rise until boiling occurs. Fig. 2a illustrates the two regimes: a nearly constant temperature near the bottom of the dewar where heat can be transferred by convection; with the other regime above, where boiling occurs, but at a temperature 0.1 K above that expected from a vapour-liquid equilibrium state alone. Presumably, the empty larger dewars have turbulent flow conditions which can transport more heat than laminar flow found in the smaller diameter dewar or the larger dewar with waveguide.

Conclusion

The temperature distribution in dewars of liquid nitrogen appears to be governed by vapour-liquid equilibrium and convection. The first is dominant in dewars of 7.5 cm diameter at high heat fluxes (3 W) into the body of the liquid. In dewars of 15 cm diameter and in smaller dewars with low heat flux, the second heat transfer mechanism is dominant.

Temperature fluctuations increase with increasing heat flux, and increasing dewar diameter, and are higher near the surface than in the bulk of the liquid. There is some critical dimension between 7.5 and 15 cm, below which temperature fluctuations become less than 1 mK.

Liquid nitrogen in a liquid nitrogen shielded dewar tends to come to an isothermal condition with a temperature .15 K to .3 K above the surface temperature, for heat inputs up to 2 W and liquid depths between 50 and 70 cm. Approximately half the liquid is superheated and the remainder subcooled.

This condition can be used to provide an extensive and cheap thermal environment for large experimental apparatus that is isothermal to ±10 mK and at a temperature that may be held over many hours to ± 5 mK.

Y. Horio in his Southampton University MSc thesis (1975) on the vertical temperature distribution in LNG storage tanks, observed an isothermal vertical temperature profile to within ± 1 K over a depth of 30 m.

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