TEMPERATURE MEASUREMENT IN A SOLUTION CRYOSTAT WITH A NUCLEAR THERMOMETER

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Temperature can be measured on the basis of the nuclear-resonance signal amplitude or the spin-lattice relaxation time by observing in a magnetic field the free precession induced in platinum nuclei by a resonant radio-frequency wave train. The results of measurements in a cryostat with a solution of He^3 in He^4 show that a nuclear thermometer of pure platinum is suitable for thermometry purposes, but measurements with it should not be carried out more frequently than every 10–20 seconds.

He^3 solution cryostats make it possible to obtain a temperature of several millidegrees, which hitherto has been measured by determining the magnetic susceptibility of cerium-magnesium nitrate (CMN). In this temperature region, however, the magnetic temperature of CMN may differ noticeably from the thermodynamic temperature, and therefore experiments were performed to determine the temperature with the aid of the magnetic resonance of the nuclei Pt^{195} (35.3%). The parameters were as follows: nuclear spin $I = 1/2$, nuclear magnetic moment $\mu = 3.06 \times 10^{-24}$ erg/G, $\gamma = 915.3$ Hz/G, and $T\tau_1 = 0.3$ sec-deg, where τ_1 is the spin-lattice relaxation time, and the nuclear-spin relaxation time is $\tau_2 = 0.001$ sec. The lower part of the solution cryostat and the system for observation of the nuclear resonance of platinum are shown in Fig. 1.

The nuclear-thermometer pickup 2 was made up of platinum-foil plates 20 μ thick, insulated from one another and placed perpendicular to the magnetic field H_0. The pickup contained 1 g of platinum and had the form of a cylinder of 1 cm diameter and 1 cm height. The constant field $H_0 = 470$ G was produced by Helmholtz coils of superconducting wire with "frozen" superconducting current. The field $\tilde{H}$ with amplitude ~0.2–1 G was applied in the form of a train of waves with duration t = 0.3 msec and resonant frequency f = 430 kHz, and rotated the magnetization vector of the Pt^{195} nuclei through an angle $\theta = 180^\circ \times \gamma Ht \approx 10–50^\circ$. Precessing around H_0, the magnetization vector induced in the receiving coil a nuclear-resonance signal. At n = 240 turns and Q = 30 in circuit 3 with the coil, the nuclear-resonance signal at the amplifier input was $\mathcal{E} = 40T^{-1} \sin\theta$ [μV].

The nuclear-resonance signal was amplified by resonant amplifier 4 and fed to a long-persistance oscilloscope. To prevent overloading, the amplifier was automatically shunted during the time of application of the train. At an amplifier bandwidth $\Delta f = 6$ kHz, the total noise referred to the input of the amplifier was 0.3 μV, and therefore it was possible to measure the temperature at T = 4°K with accuracy up to 4% for a 90° train ($\theta = 90^\circ$), and with an accuracy of about 30% for a 6° train. At lower temperatures, the measurement accuracy was higher and was determined by the accuracy with which the readings were picked off from the long-persistence oscilloscope, namely several per cent.

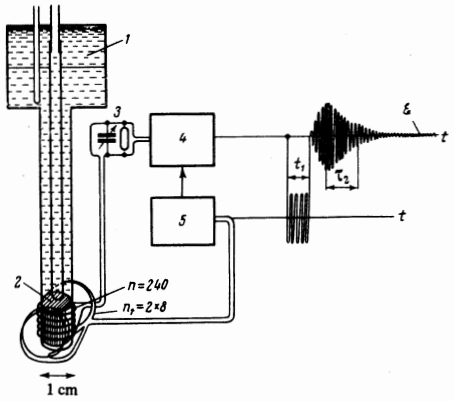

FIG. 1. Diagram of platinum nuclear thermometer: 1–solution chamber, 2–pickup of nuclear thermometer, 3–resonant circuit with coil, Q = 30; 4–resonant amplifier, $K = 3 \times 10^4$, f = 430 kHz, $\Delta f = 6$ kHz; 5–wave-train generator, f = 430 kHz, J = 0–1A.

Figure 2 shows photographs of the screen of the long-persistence oscilloscope–at $T \approx 1$°K on the top and at $T \approx 0.01$°K on the bottom. The damping time τ_2 of the nuclear-resonance pulse was 1 msec. Figure 3 shows a calibration of the thermometer placed in liquid helium, against the vapor tension of He^4 above 1.6°K and against the vapor tension of He^3 below this temperature.

It should be noted that at first attempts were made to use commercial platinum or samples of not very pure platinum–in such samples the depth of penetration of the alternating field is higher. But the attempts were not successful. We did not observe a temperature dependence of the signal in the form $\mathcal{E} \propto 1/T$. The platinum used in the described experiments was of maximum purity, such as is used for standard platinum resistance thermometers. For a foil of thickness 20 μ, the resistance ratio was $R(300°K)/R(1.2°K) = 1000$.

Another independent but no less accurate method of measuring the temperature was the use of the ratio $T\tau_1 = 30$ msec/deg, which follows from the Korringa theory[1] and from the experiments of Walstedt et al.[2]. The top half of Fig. 4 shows a photograph taken from the screen of a long-persistance oscilloscope used to

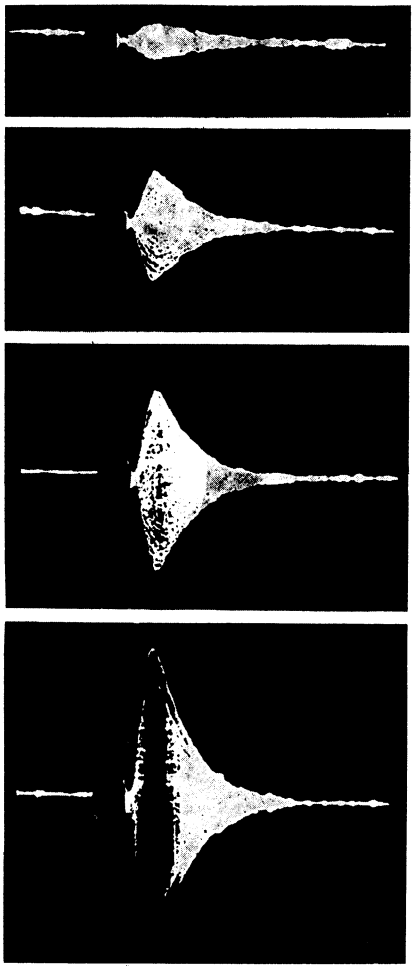

FIG. 2. Photographs of signals from the screen of the long-persistence oscilloscope, obtained by varying the temperature from T = 2°K (top) to T = 0.01°K (bottom).

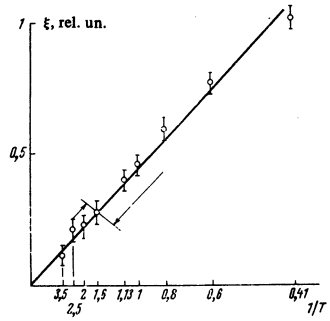

FIG. 3. The dependence of the nuclear signal on the reciprocal temperature. The numbers on the abscissa axis—degrees Kelvin.

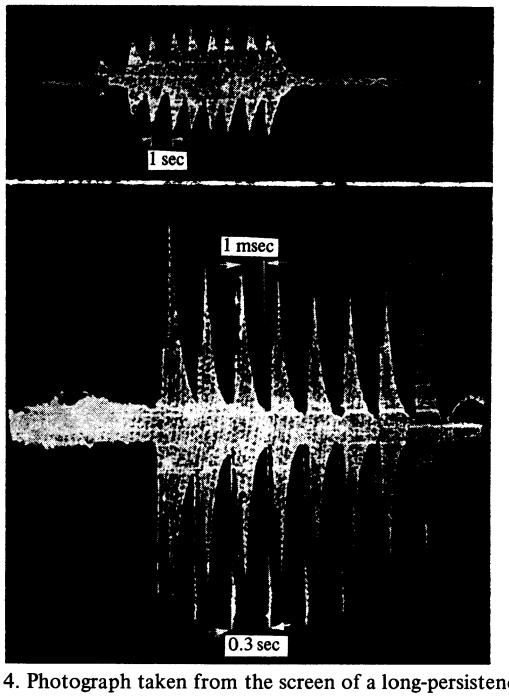

FIG. 4. Photograph taken from the screen of a long-persistence oscilloscope, obtained for a series of a train of waves of duration 0.3 msec following each other in intervals of 1 sec (upper photograph) and 0.3 sec (lower photograph); the start of the registration of the signal from each succeeding train is shifted.

register nuclear-resonance signals obtained by applying a series of wave trains following one another in intervals of 1 sec. The start of the plot of each succeeding signal was shifted on the oscilloscope screen. Directly before the application of the series of trains, a generator operating at a frequency close to resonant was applied for 10 sec to the exciting coils. This caused demagnetization of the platinum atoms followed by magnetization with a time τ_1, i.e., in accordance with the relation $A = A_0(1 - \exp[-t/\tau_1\tau])$ or $\ln[A_0/(A_0 - A)] = t/\tau_1$. Plotting $\ln[A_0/(A_0 - A)]$ against t, we can determine τ_1, and consequently also the temperature. For the given photograph we have T = 0.017°K with accuracy to 10%.

Measurement of the temperature with a nuclear thermometer by using a train of waves of duration $t_1 = 0.3\tau_2$ is preferable to the single half-wave pulse used in[3] and to the continuous application of the resonant frequency used by Osgood and Goodkind[3]. To effect rotation through the same angle θ it is necessary in the former case of use a field $\tilde{H}_I$ which is stronger by $2\nu t_1 = 260$ times than our field, and the heat release in this case will also be $(1/2)\tilde{H}_I^2 T/\tilde{H}^2 t_1 = \tilde{H}_I/\tilde{H} = 260$ times larger. When the frequency is applied continuously, the signal level is equivalent to the level occurring upon rotation through an angle θ_{II}, with $\sin\theta_{II} \approx \pi\gamma\tilde{H}\tau_2$, i.e., the same signal will be obtained at a field $\tilde{H}_{II} = t_1\tilde{H}/\tau_2 = 0.3\tilde{H}$. However, for even a single measurement during a time $t_2 = 3$ sec, the average power released in measurements using a wave train will be smaller by a factor $\tilde{H}_{II}^2 t_2/\tilde{H}^2 t_1 = 900$. In addition, when the continuous measurement method is used, the thermometer is always overheated compared with the medium, owing to the eddy currents. Although measurement with the aid of trains leads to a temporary overheating of the thermometer and of the ambient, the measured amplitude corresponds to the value of the magnetic moment prior to the application of the train, i.e., to the true temperature at the instant of the start of the measurement.

That the heating produced by the measurement is important can be seen from the lower curve of Fig. 4, when the wave trains were applied at T = 0.03°K in intervals of 0.3 sec. The first wave train produces a signal 20% larger than the succeeding train. This means that the system of nuclei is heated during the

time of application of the train by $\Delta T = 0.2T = 6 \times 10^{-3}$°K. Since the nuclear specific heat in the field H = 470 G is equal to

$$C = \frac{N\mu^2(I+1)H^2}{3kIT^2} = 1.8 \cdot 10^{-2} \text{ erg/deg}$$

where $N = 1.1 \times 10^{21}$ is the number of Pt^{195} atoms in the sample, and k is Boltzmann's constant, the nuclear system absorbed $\sim 10^{-4}$ erg as it became heated. This heat was then transferred to the lattice and to the platinum electrons, with their relaxation time $\tau_1 = 1$ sec.

The question of establishment of thermal equilibrium at low temperatures is particularly important. The volume specific heat of platinum, $C_1 = 7.5 \times 10^{-4} T$ [J/cm³deg] is smaller by a factor 330 than the specific heat of the solution of He^3 in He^4, which equals $C_2 = 0.25\ T$ [J/cm³deg], and therefore the time of establishment of equilibrium between the platinum and the solution can be determined from the formula $\tau = RC_1\delta/2$, where $\delta = 2 \times 10^{-3}$ cm is the thickness of the platinum, and R is the Kapitza jump between the platinum and the solution. According to measurements by Zinov'eva[4], for copper and stainless steel at $T < 0.4$°K we have $R = 50\ T^{-3}$ deg-cm²/W. If we assume also for platinum the same value of R, then at T = 0.003°K the time is $\tau = 4$ sec, and at higher temperatures it is much shorter, since $\tau \sim 1/T^2$. Establishment of equilibrium in the solution is helped by the constant downward flow of He^3 atoms from the solution boundary, around the sample, and then up along the tube. In addition, the thermal conductivity of the solution is appreciable and increases with decreasing temperature: $\kappa = 2.5 \times 10^{-6}\ T^{-1}$ W/cm-deg, and therefore the time of establishment of equilibrium in the solution by thermal conductivity is given by $\tau = C_2 l^2/\kappa\pi \sim T^2$, and equals $\tau = 4$ sec if the characteristic dimension in the liquid is $l = 2$ cm and if T = 0.01°K; at lower temperatures it is even smaller.

It can thus be concluded that a platinum nuclear thermometer is suitable for temperature measurements in solution cryostats, but the measurements must be performed at intervals not shorter than 10–20 sec.

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[4] K. N. Zinov'eva, Zh. Eksp. Teor. Fiz. 60, 2243 (1971) [Sov. Phys.-JETP 33, 1205 (1971)].

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