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Abstract

The construction and performance of a simple, inexpensive cryostat of the "cold-finger" type is described. It is specifically intended for Mössbauer measurements between -195 and +50°C; the accuracy is better than ± 0.5 deg C.

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Introduction

The various principles of cryostats for Mössbauer spectrometers have been described in a series of review articles e.g.[1, 2]. Two types are most commonly used: "cold-gas-stream" and "cold-finger" cryostats. The former type is especially suited where a great accuracy, better than ± 0.1 deg C, is required, which may be the case in studies of phase transitions. A similar accuracy can only be obtained with an advanced cold-finger cryostat[3], but simpler types may offer the advantage of better long term stability.

In a cold-finger cryostat a metal rod with high thermal conductivity connects the sample to be cooled with a liquid gas in a Dewar vessel; liquid nitrogen is normally used as cooling agent since it is easily available, has a low boiling point (- 196°C at 1 atm.), and a relatively large heat of evaporation (38.6 kcal/l). The temperature can be regulated with a small electric heater placed near the sample. For measurements that do not require a better accuracy than ± 0.5 deg C, a simple cold-finger cryostat is the easiest and cheapest solution; by using exchangeable thermal bridges it is possible to cover the temperature range -195 - + 50°C with a modest consumption of liquid nitrogen. A cryostat of this type is described below.

Design

The cryostat is supported by a table (cf. fig. 1); under the table is placed a Dewar vessel into which the cold finger dips. The cold finger is made of a solid copper cylinder (250 mm x 30 mm ø); it is screwed on an exchangeable thermal bridge (90 mm x 16 mm ø), which in its turn is screwed on a sample house made of solid copper. Because of the small relative velocities between absorber and source used in the recording of Mössbauer spectra, it is essential that no vibrations - e.g. caused by boiling nitrogen - are transferred to the sample; for this reason the suspension of the sample house in Mössbauer cryostats is a crucial point. In the present case, the sample house is supported by a cross of monel tubing (o.d. 3 mm) which rests in tracks in a lucite ring.

In the sample house a grove is milled for a heating coil made of manganine wire (o.d. 0.70 mm; 1.17 ohms/m) with a resistance of 5.0 ohms. In horizontal borings two temperature sensors are placed, namely a thermocouple (chromel-alumel) and a resistance thermometer (platinum, 100 Ω at 0°C; Degussa type P7). All electric connections are led through holes in the lucite ring which supports the monel cross. The sample holder proper is placed in a horizontal boring closed at both ends with windows of Mylar foil (thickness 50 µ).

The sample house is insulated with a housing of urethane foam (urethane foam has a somewhat larger density than styrofoam, but insulates equally well and is easier to manufacture). The upper part of the housing can be removed for changing of samples.

With the materials used here, the thermostat can only be operated safely up to about 50°C, but in principle such systems can be used at several hundred degrees.

Temperature Regulation and Measurement

The resistance thermometer, connected in series with a variable resistor (10 turns, 200 ohms), forms one branch in a Wheatstone bridge, the other three branches being 200-ohm precision resistors (cf. fig. 2). The error signal is passed through a phase-sensitive amplifier and used to trigger a relay which "on - off" regulates the voltage to a transformer. The transformer supplies power to the heating coil; a voltage of about 10 V is normally suitable.

The resistance of the resistance thermometer varies between about 20 ohms at -195°C and about 120 ohms at + 50°C; a desired temperature is obtained by adjusting the variable resistor to a value which makes the total resistance in the regulating branch of the bridge 200 ohms. The accuracy of the temperature regulation is essentially determined by three factors: (1) the temperature dependence of the resistance thermometer, (2) the sensitivity of the amplifier and the relay, and (3) the thermal conductivity of the sample house. As shown below, however, the last factor can be ignored in the present case. The resistance of the thermometer increases by the order of 0.4 ohms per deg C; the sensitivity of the detecting system was determined at fixed temperatures, by varying the variable resistor, to be about 0.2 ohms; therefore the sensitivity of the regulating system itself is about 0.5 deg C.

In principle, the setting of the variable resistor is a measurement of the temperature, but a more accurate value of the actual temperature of the sample is measured with the thermocouple, which is used in a conventional way with a reference bath and a galvanometer.

The whole set-up is shown in a photograph, fig. 3.

Experimental Data

In table 1 various bridge materials are proposed, and it appears that only three thermal bridges, aluminium, iron, and stainless steel are necessary to cover the temperature interval -190 - +50°C. With these materials the liquid nitrogen consumption is below 0.3 l/h.

The temperature stability was investigated for three representative temperatures, -170, -100, and -30°C; the output from the thermocouple was registered on a recorder, and in all cases the variations were below ± 0.01 mV, corresponding to a temperature stability better than ±0.5°C. Since this corresponds to the sensitivity of the regulating system, we conclude that the time constant of the heat transport in the sample house is negligible.

The cryostat has been used in Mössbauer investigations of after-effects of the ^{57}Co(EC)^{57}Fe-decay in hexacyanides. The transmission of the 14.4 keV γ-quanta through the cryostat proper is better than 80%. Spectra of absorbers recorded with a source of ^{57}Co in palladium show linewidths of the same magnitude as those reported in the literature, so it seems that vibrations of the sample have been avoided.

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3) F. van der Woude and G. Boom, Precision (0.1°C) Vacuum Cryostat and Furnace for Mössbauer Experiments. Rev. Sci. Instrum. 36 (1965) 800-802.

4) e. g. Dwight E. Gray (editor), American Institute of Physics Handbook. 3rd edition (McGraw-Hill, New York, 1972).

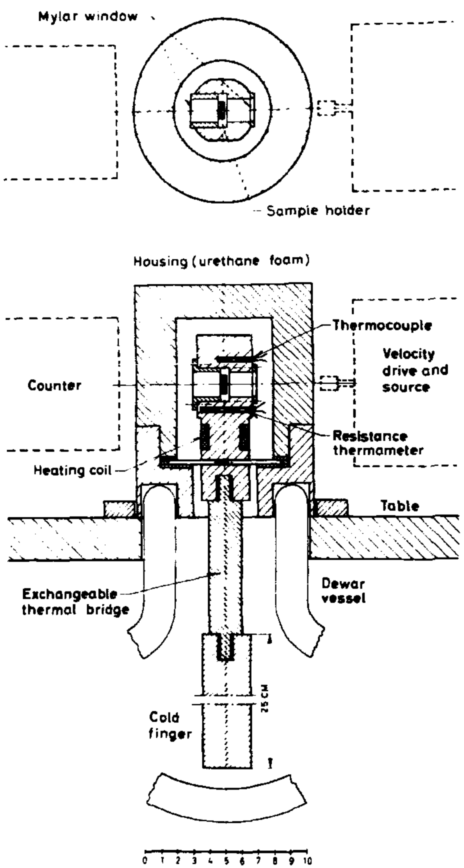

Fig. 1. A vertical cut through the Mössbauer cryostat (slightly simplified). The counter and the velocity drive with source, drawn in dashed line, are arranged for an absorber measurement.

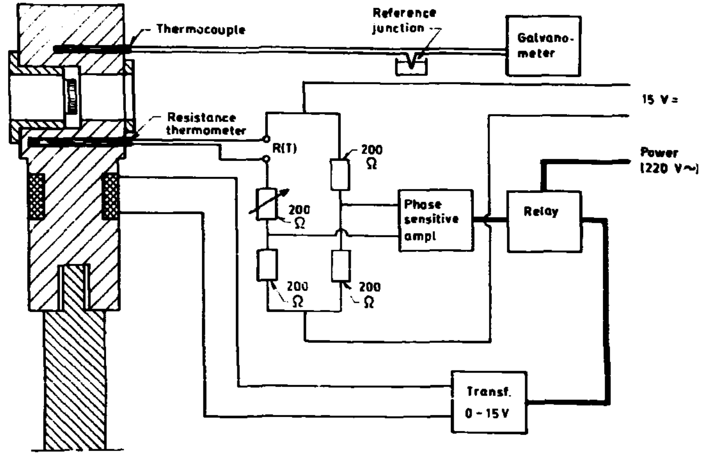

Fig. 2. Block diagram of the temperature regulating and measuring system.

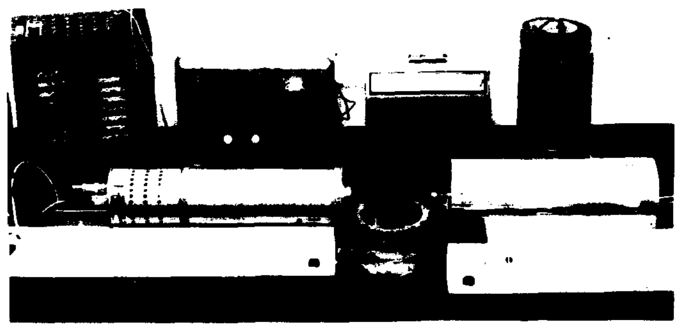

Fig. 3. A photograph of the Mössbauer set-up with the housing of the cryostat removed.

Table 1

Examples of thermal-bridge materials

Bridge material with thermal conductivity at $0^{o}C$ [4)] $\left(\frac{\text{Watts}}{\text{meter} \cdot \text{deg C}}\right)$	Useful temperature interval (deg C)
Copper 4.01	-195 - -175
Aluminium 2.35	-190 - -120
Brass ~ 1.20	-160 - -100
Iron 0.835	-120 - -50
Stainless steel ~ 0.14	-50 - +50

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