Technical University of Denmark

A simple thermostat based on a temperature-regulated gas stream

Forskningscenter Risø, Roskilde; Fenger, J.

Publication date:
1974

Document Version
Publisher's PDF, also known as Version of record

Risö-M - 1694

Danish Atomic Energy Commission

Research Establishment Risö

Risö-M

CHEMISTRY DEPARTMENT

A Simple Thermostat Based on a Temperature-regulated Gas Stream

by

E.B. Andersen and J. Fenger

March 1974

Available on request from: Library of the Danish Atomic Energy Commission (Atomenergikommissionens Bibliotek), Risö, DK-4000 Roskilde, Denmark.
Telephone: (03) 35 51 01, telex: 5072

A. E. K. Risø

Risø-M- 1694

Risø-M- 1694

Title and author(s)

A Simple Thermostat Based on a Temperature-regulated Gas Stream

by

E.B. Andersen and J. Fenger

Date March 1974

Department or group

Chemistry Department

Group's own registration number(s)

6 pages + 0 tables + 2 illustrations

Abstract

The construction and performance of a thermostat of the "Gas-Stream" type are described. It was developed for Mössbauer measurements, but can equally well be used in connection with other types of spectrometry. In the present form it has been used in the temperature range -150 - +250°C, with a regulating sensitivity of ±0.5°C.

Copies to

Library 100

Standard distribution

Abstract to

Available on request from the Library of the Danish Atomic Energy Commission (Atomenergikommissionens Bibliotek), Risø, Roskilde, Denmark.
Telephone: (03) 35 51 01, ext. 334, telex: 5072.

Fi 25-204

Introduction

Cryostats for use in Mössbauer spectrometry, [e.g. 1] can roughly be divided into two types:

In 'Cold-finger cryostats' a heat-conducting rod connects the sample holder with a cold reservoir, e.g. a bath of liquid nitrogen; the temperature can be regulated with an electrical heater placed near the sample, [e.g. 2]. This type of cryostat is easy to build and it has a low consumption of nitrogen; the temperature stability can in advanced versions,[e.g. 3] be $\pm 0.1^{o}C$. On the other hand it it not well suited for work in large temperature ranges extending above room temperature.

In 'Gas cryostats' the sample holder is in thermal contact with a temperature-regulated gas. Different temperatures can be obtained by various means, e.g. by mixing two gases of different temperatures [4] or by heating a cold gas appropriately. 'Gas cryostats' are generally more complicated and expensive than 'cold-finger cryostats', but they are well suited in experiments which require measurements both at low and high temperatures in an unchanged geometry.

The present report describes the construction of a cryo-thermostat based on the gas-stream principle. It was intended for determinations of formal Debye temperatures in the mineral arfvedsonite [5]; this determined the temperature range -150 - $+250^{o}C$, which can, however, easily be extended by minor modifications. In the design mechanical stability has been stressed more than accuracy in the temperature regulation. Although the set-up was built for Mössbauer measurements, it may also be used in other types of spectroscopy.

Design

The principle in the cryo-thermostat is shown in fig. 1. A cold nitrogen gas is produced by boiling of liquid nitrogen in a 50 l Dewar vessel (Siegtal type TS50), in which is immersed a heater made from five 20 Ω/4 W resistors in series. To protect against over-pressure the Dewar vessel is equipped with a safety valve. The cold nitrogen gas is passed through an insulated quartz tube with a heater coil in order to give the desired temperature. A safety switch, activated by a thermocouple, protects against overheating in case of failure in the temperature regulation; this heating system (Varian type V-4547) was borrowed from an ESR-spectrometer. The gas is then led to a T-tube, which on one branch has a modified Edwards union for introducing a platinum resistance thermometer (Degussa type P7, 100 Ω at $0^{\circ}C$). The other branch goes to the sample holder.

The resistance thermometer, connected in series with a variable resistor (10 turns, 100 Ω), forms one branch in a Wheatstone bridge, the other three branches being 220 Ω precision resistors. The error signal is passed through a polarity-sensitive amplifier and used to trigger a relay which 'on-off' regulates the voltage to the transformer in the heating system.

The resistance of the resistance thermometer varies between about 40 Ω at $-150^{\circ}C$ and about 200 Ω at $+250^{\circ}C$; 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 220 Ω. The accuracy of the temperature regulation of the gas stream is essentially determined by two factors: (1) the temperature dependence of the resistance thermometer, (2) the sensitivity of the amplifier and the relay. The resistance of the thermometer increases by the order of 0.4 Ω per deg. C; the sensitivity of the detecting system was determined at fixed temperatures, by varying the variable resistor, to be about 0.2 Ω; therefore the sensitivity of the regulating system itself is about 0.5 deg. C.

A vacuum insulating chamber made of brass with a sample holder for Mössbauer absorbers is shown in fig. 2. The sample is a thin powder layer clamped in a perspex box; it is placed in the middle of a thick-walled brass tube around which is wound a cobber tube (i.d. 4 mm; o.d. 6 mm). Through this tube the thermostated nitrogen gas is passed; in order to allow transmission of the radiation through the vacuum chamber, this is furnished with two 50 μ mylar windows. The temperature is monitored with a thermocouple.

Performance

The temperature range which can be covered by the set-up is in the lower end determined by the quality of the insulation. Because of the relatively high Debye temperature of the sample [5] very low temperatures were not essential. With a primitive insulation partly made of available vacuum tubes, partly of rubber from hoses (Armaflex) temperatures down to -150°C could be obtained with a liquid-nitrogen consumption of about 100 l/d. At -100°C the nitrogen consumption is about 20 l/d. The electrical effect necessary to evaporate these amounts is 250 and 50 watts respectively; it can be adjusted via the voltage over the heater in the Dewar.

For temperatures above room temperature, the cold nitrogen stream can be replaced by pressurized atmospheric air; a limit to the temperature which can be obtained is only set by the capacity of the heater and the stability of materials used in the construction. Surprisingly the perspex box containing the sample could, under the experimental conditions, stand temperatures up to 250°C; this corresponds to a heater effect of about 30 watts, with a flow of about 10 l/min.

References

1) N. Benczer-Koller and R.H. Herber. In: Chemical Applications of Mössbauer Spectroscopy. Edited by V.I. Goldanskii and R.H. Herber (Academic Press, New York, 1968) 134-158.

2) L.A. Frees and J. Fenger, A Simple Mössbauer Cryostat Based on the "Cold-Finger" Principle. Risø-M-1662 (1973) 8 pp.

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) J. Fenger, A Temperature Regulating System for Use with an Electron Spin Resonance Spectrometer. J. Sci. Instrum. 42 (1965) 904-905.

5) E.B. Andersen, J. Fenger, and J. Rose-Hansen, Determination of Fe^{2+}/Fe^{3+}-Ratios in Arfvedsonite by Mössbauer Spectroscopy, to be published.

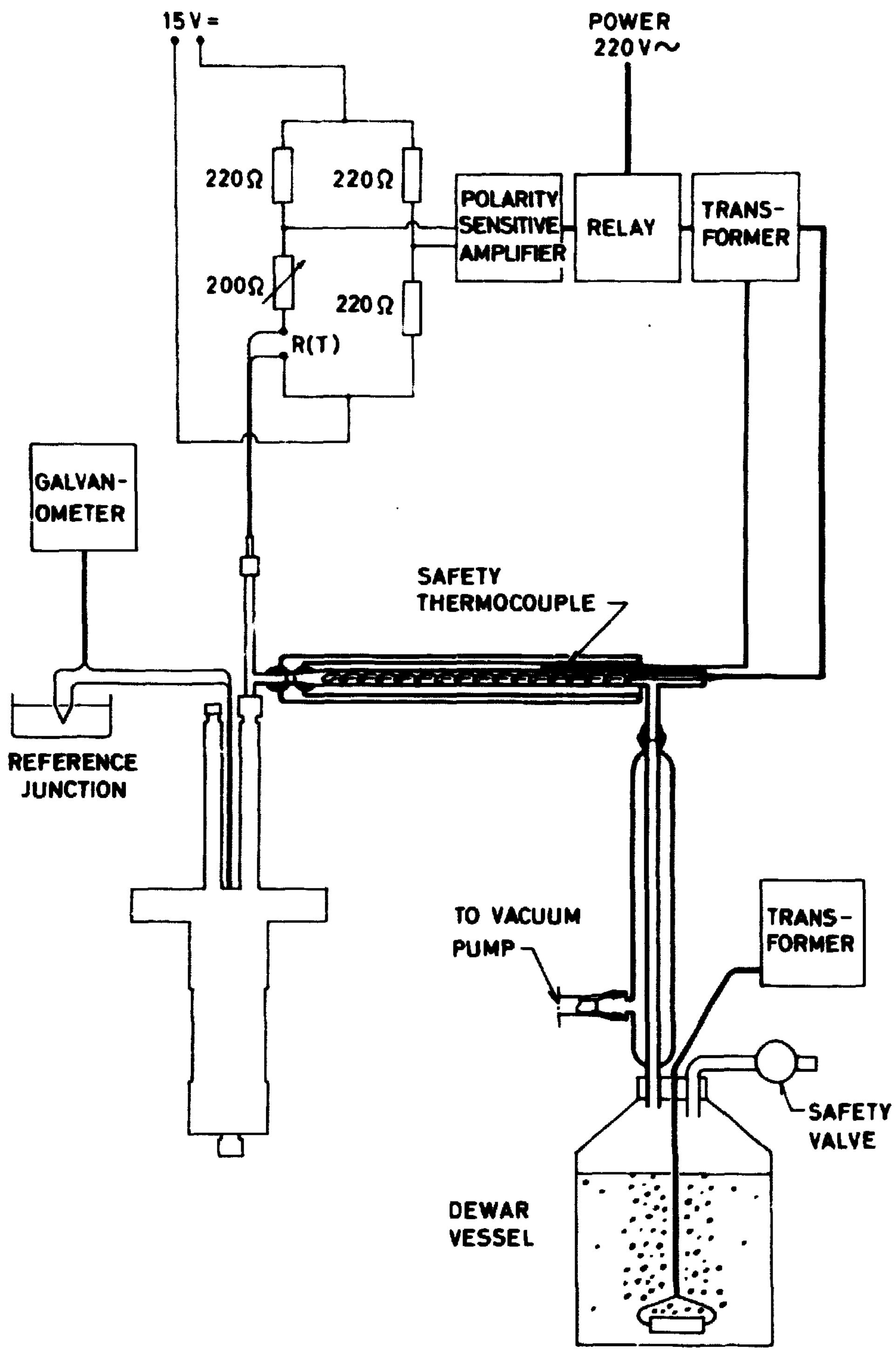

Fig. 1. Simplified diagram of the temperature regulating and measuring system. The various parts are not drawn to scale.

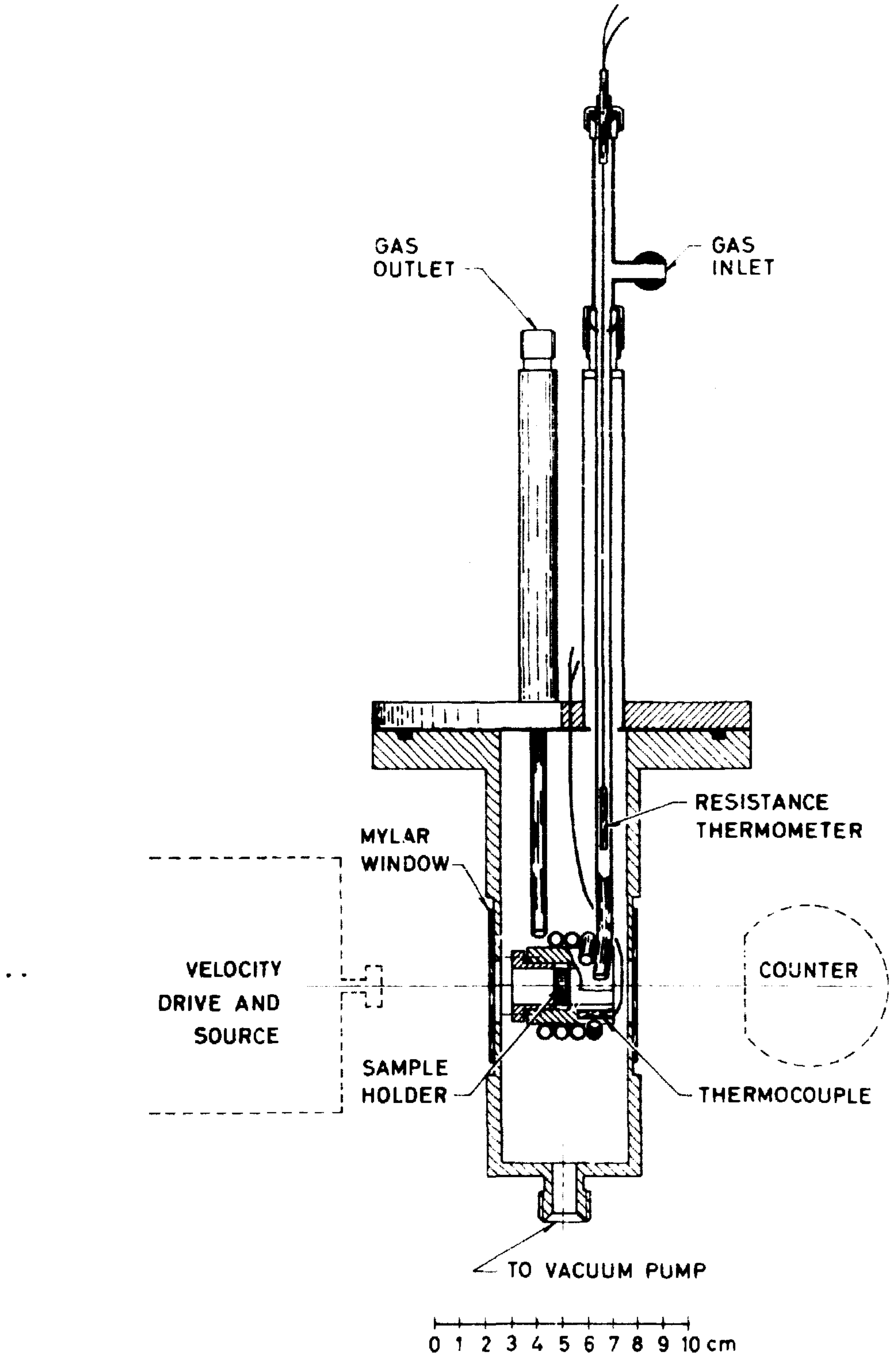

Fig. 2. A cut through a vacuum chamber with sample holder designed for Mössbauer measurements. Typical positions of the source and counter are drawn in dashed line. Collimators, mechanical supports, etc. are omitted.

ISBN 87 550 0246 3