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# Description of a differential setup for relaxation microcalorimetry

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ABSTRACT

A specific heat measuring instrument, with the capacity for the application of magnetic fields up to 9 T, resorting to microcalorimetry chips from the company Xensor Integration, has been successfully assembled and its functioning specifications are reported in the current paper.

With this instrument is it possible to perform specific heat measurements with applied magnetic fields up to 9 T in milligram samples. This offers our group the possibility to calculate the actual adiabatic temperature change of a material, as well as providing reliable and precise information on any phase transition that may be influenced by the application of a magnetic field.

# Dispositif différentiel pour la microcalorimétrie des contraintes

*Mots clés :* microcalorimétrie ; effet magnétocalorique ; mesures des contraintes ; chaleur massique ; puces Xensor

## 1. Introduction

Since the beginning of magnetocaloric research, technically inaugurated by Brown in 1978 (Gschneidner and Pecharsky, 2008), the method most generally used for the evaluation of the magnetocaloric potential of a given material is the application of Maxwell's thermodynamic equation to a set of appropriately measured magnetic isotherms, so as to calculate the magnetic entropy change (Debye, 1926). Controversies regarding the validity of this equation aside, the usual alternative to this method is the calculation of the adiabatic temperature change, which translates the actual cooling and heating of a material.

The calculation of this temperature change relies on isofield specific heat measurements, as well as the same magnetization measurements as required by Maxwell's thermodynamic equation mentioned above (Tishin and Spichkin, 2003). Unfortunately this calculation is usually made difficult by the lack of a

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**Nomenclature**

| | |
|---|---|
| $V$ | Thermopile excitation |
| $t$ | Time |
| $A$ | Constant (does not play a role in our calculations) |
| $\tau$ | Relaxation time |
| $C$ | Heat Capacity |
| $\kappa$ | Thermal conductivity between the sample and its environment |
| $P$ | Heating power supplied |
| $T_1$ | Initial temperature |
| $T_2$ | Temperature after heating |

commercial measurement system that would allow for reliable specific heat measurements with an applied magnetic field.

Typically, to overcome this issue, many research groups world wide resort to assembling their own isofield specific heat measuring equipments. Among the most recent examples we may cite the setup described by Marcos et al. (2003), consisting of an insert that can be fitted to any cryostat with the capacity to generate a magnetic field. This setup resorts to thermobatteries which give a voltage output in response to the heat exchange with the measured sample, which can then be read and interpreted as numerical value.

One other example may be observed in Korolev et al. (2005), although this system has been specifically designed to measure magnetic colloids. Instead of using a permanent magnet it is designed as a microcalorimetry cell placed between the two poles of an electromagnet so as to generate a (low intensity) magnetic field.

The setup described by Kuepferling et al. (2007) on the other hand resorts to commercial Peltier cells, a thermoelectric device made of a series of junctions of conductors with different thermoelectric power, acting as both sensors and actuators, being in this way able to achieve strict isothermal conditions. However this setup is highly dependent on accurate calibration of the Peltier cells.

Under this perspective the microcalorimetry chips from the company Xensor Integration have gained increasing relevance, due to their precision, practicality and relatively small price, as exemplified by Morrison et al. (2008) or Minakov et al. (2005a, 2005b, 2007), making them a very attractive and promising tool for the planning and assembly of such calorimeters.

We now report the design and construction of an experimental setup that would allow for specific heat measurements under high magnetic fields, resorting to such microcalorimetry chips. We have adopted a two chip setup in our equipment which enables us to easily bypass many bothersome calibration and equipment specific issues. This instrument's potential ranges well beyond the purely magnetocaloric oriented, as it can provide invaluable information regarding any phase transition where the application of a magnetic field may play a significant role.

## 2. Experimental setup

### 2.1. Cryostat

For the base of this setup we used a commercial cryostat from American Magnetics Inc. (AMI), equipped with a 9 T 2 inch bore superconducting magnet (Solenoid), its own power supply and field programmer.

This cryostat has a 36 L LHe reservoir, in direct contact with the superconducting magnet so as to keep it at a constant temperature of 4.2 K. A separate $LN_2$ reservoir, also with a capacity for 36 L, is also present so as to reduce Helium evaporation.

The original Variable Temperature Insert (VTI), fitted for transport measurements, which originally came with this equipment was removed, so as another one could be fashioned with the capacity to perform specific heat measurements. As a consequence this made it impossible to use the built-in temperature control system of this cryostat.

A schematic diagram of the cryostat is shown in Fig. 1.

The evacuated sample space is in direct contact with the liquid Helium and consists of a small 25 mm wide cylinder protected by three cylindrical shields. The first of these shields is equipped with a heater.

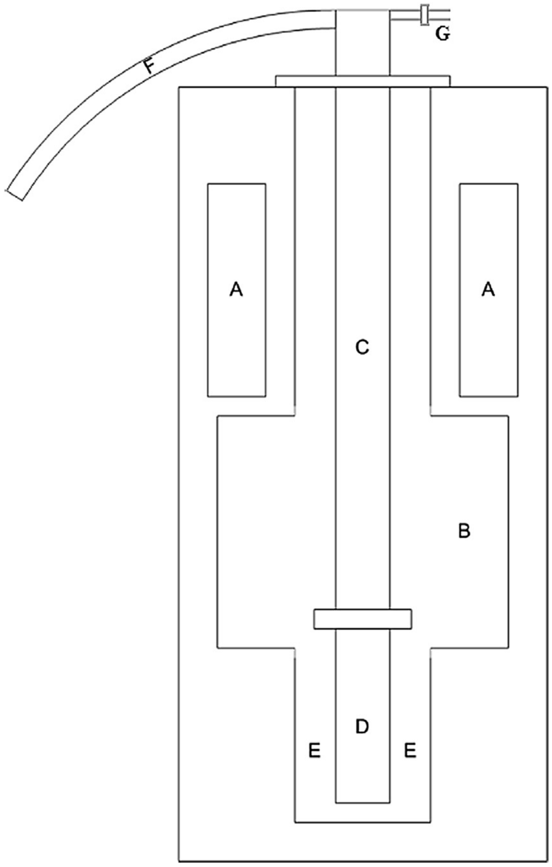

**Fig. 1 – Schematic diagram of the AMI cryostat. Legend: A) Liquid Nitrogen reservoir; B) Liquid Helium reservoir; C) Insert; D) Magnetic field center; E) Superconducting Magnet; F) Wiring conecting the insert to the rest of the measurement equipment; G) Vacuum pump.**