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AN ADIABATHIC DEMAGNETIZATION CRYOSTAT FOR DEVICE TESTING.

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ABSTRACT. Our laboratory is presently engaged in a program for the development of new detectors for ionizing radiation operating at very low temperatures which requires routine testing of materials and prototypes at temperatures under 300 mK.

For this purpose we have designed and built a small adiabathic demagnetization cryostat. The instrument, that we present here, proved to be very efficient for this kind of work and rich of virtues: it is simple and easy to use, inexpensive and reliable. As the time needed to go from 300K to 70mK is only 2 hours and sample changing being easy, the laboratory activity is somewhat speeded up.

1. - INTRODUCTION. In the last years magnetic cooling, that was abandoned in favour of He3 and He3-He4 dilution techniques, is regaining new interest(1,2).

A good reason for this is that now small efficient superconducting magnets are available at a reasonable cost.

Most of the applications deal with continuous cycle devices, but in our opinion, also one shot magnetic cooling deserves some consideration in the temperature range 30-150 mK.

In this range the temperature rise of a 100g.sample of CPA salt (Cromic potassium alum), for a typical heat link of 10**_7watt is less than 1mK/hour. This figure cannot compete with the chryogenic power of a dilution refrigerator, (50/100 mmwatt at100 mK for a small unit), but in most of the cases of interest for us the power consumption in a radiation detector is of the order of 10**-10 watts or lower.

It is then easy, by means of a feedback system, to maintain a sample at a constant temperature (within about 1mK) during several hours.

Physics of magnetic cooling is described in standard textbooks (3,4,5). In the following the thermal cycle is briefly outlined. At a starting temperature Ti, the magnetic field on the salt is isothermally increased from 0 to Bi, and consequently the salt entropy decreases as shown by the diagram of magnetic entropy of a paramagnetic salt (CPA) vs temperature in fig 1.

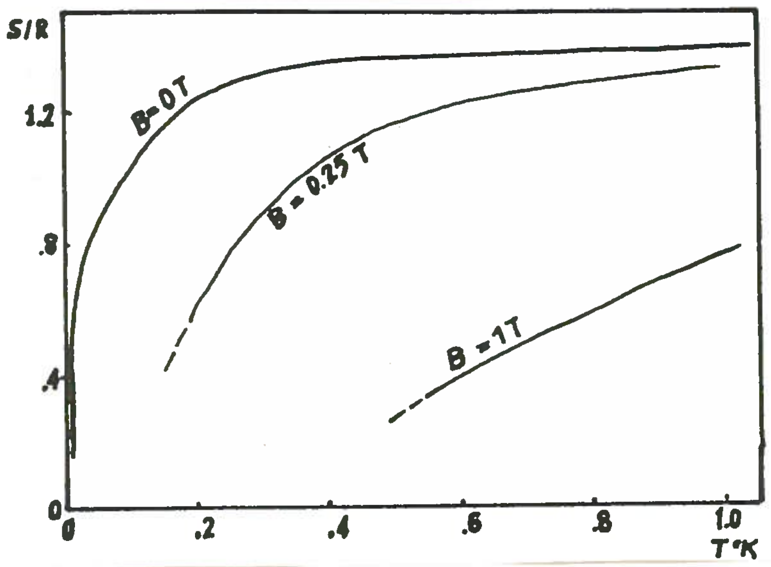

Fig.1 Entropy diagram of cpa salt.

During this phase thermal contact with the helium bath at temperature Ti is granted by the exchange gas, to be pumped away afterwards. The magnetic field is then decreased and the salt temperature more or less closely follows the isoentropic B/T curve. In our case we usually operate with a rather high starting temperature, about 1.3K, and a magnetic field of 4 Tesla, easily reached by our small superconducting solenoid. With this conditions the entropy can be reduced by a factor of about 15.

The choice of the salt is determined by the operating temperature required, in our case around 100mK. Critical properties of several suitable materials are shown in fig 2. We have thus used CPA that in this range has a maximum specific heat of about 3 J/mole.K, granting a good thermal reservoire. An alternative choice is the FAA (Ferric ammonium alum). Much lower temperatures might be obtained with CMN, (cerium magnesium nitrate), but in this case a starting temperature of 500mK is needed, that cannot be obtained by simply pumping on an He4 bath.

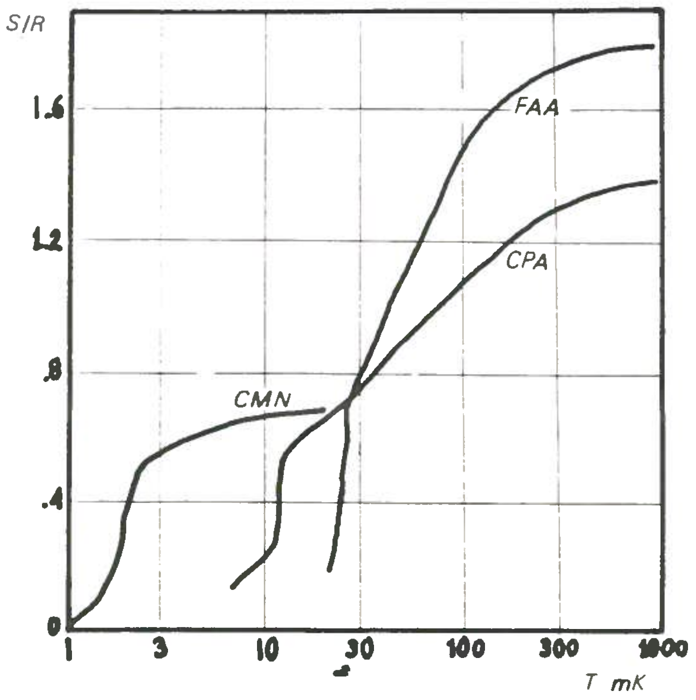

Fig.2 Entropy diagram of various paramagnetic salts.

A general view of the apparatus is outlined in fig3. In fig4, the diagram shows times needed for the various phases of a complete thermal cycle.

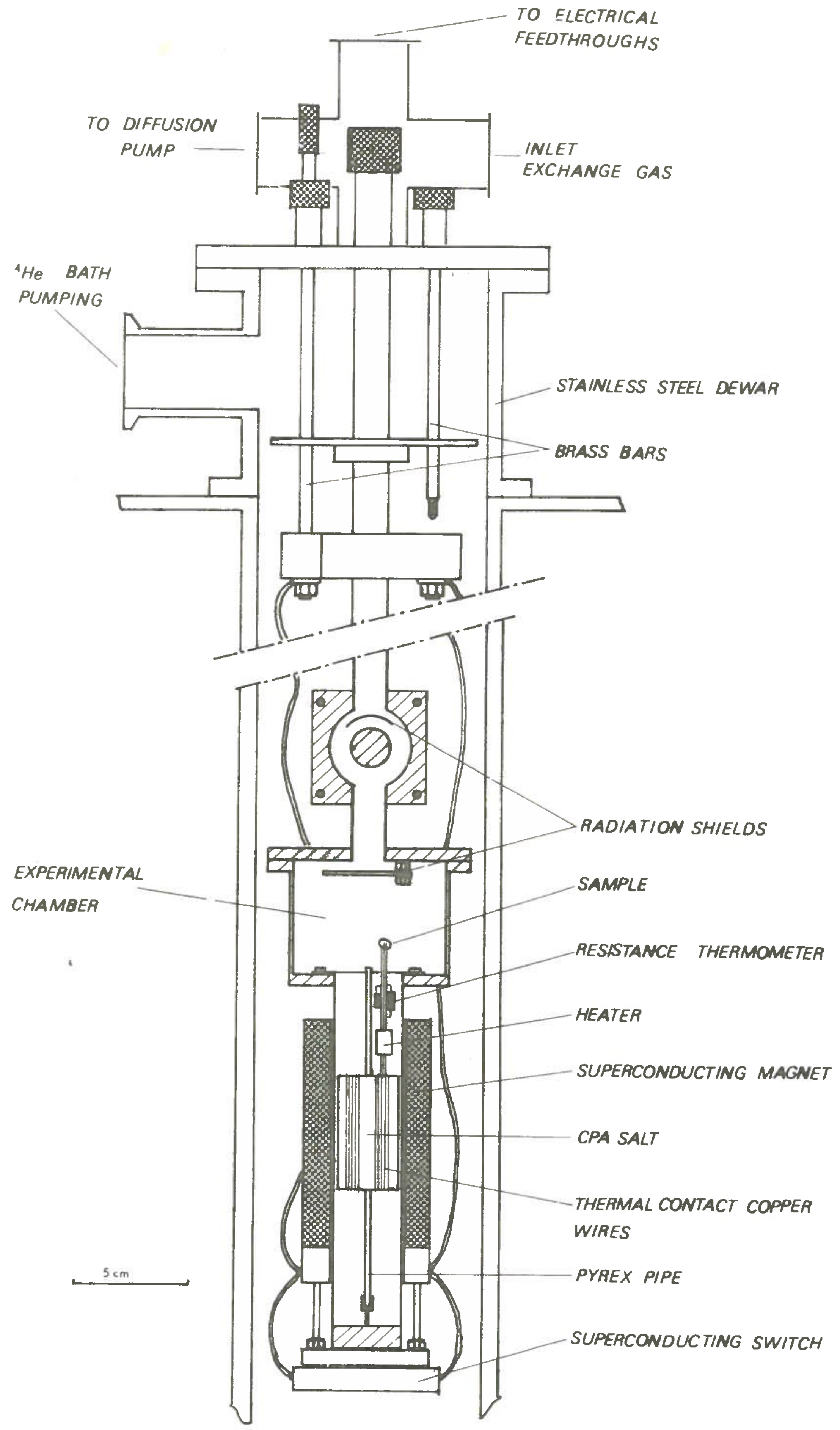

Fig.3 View of Apparatus

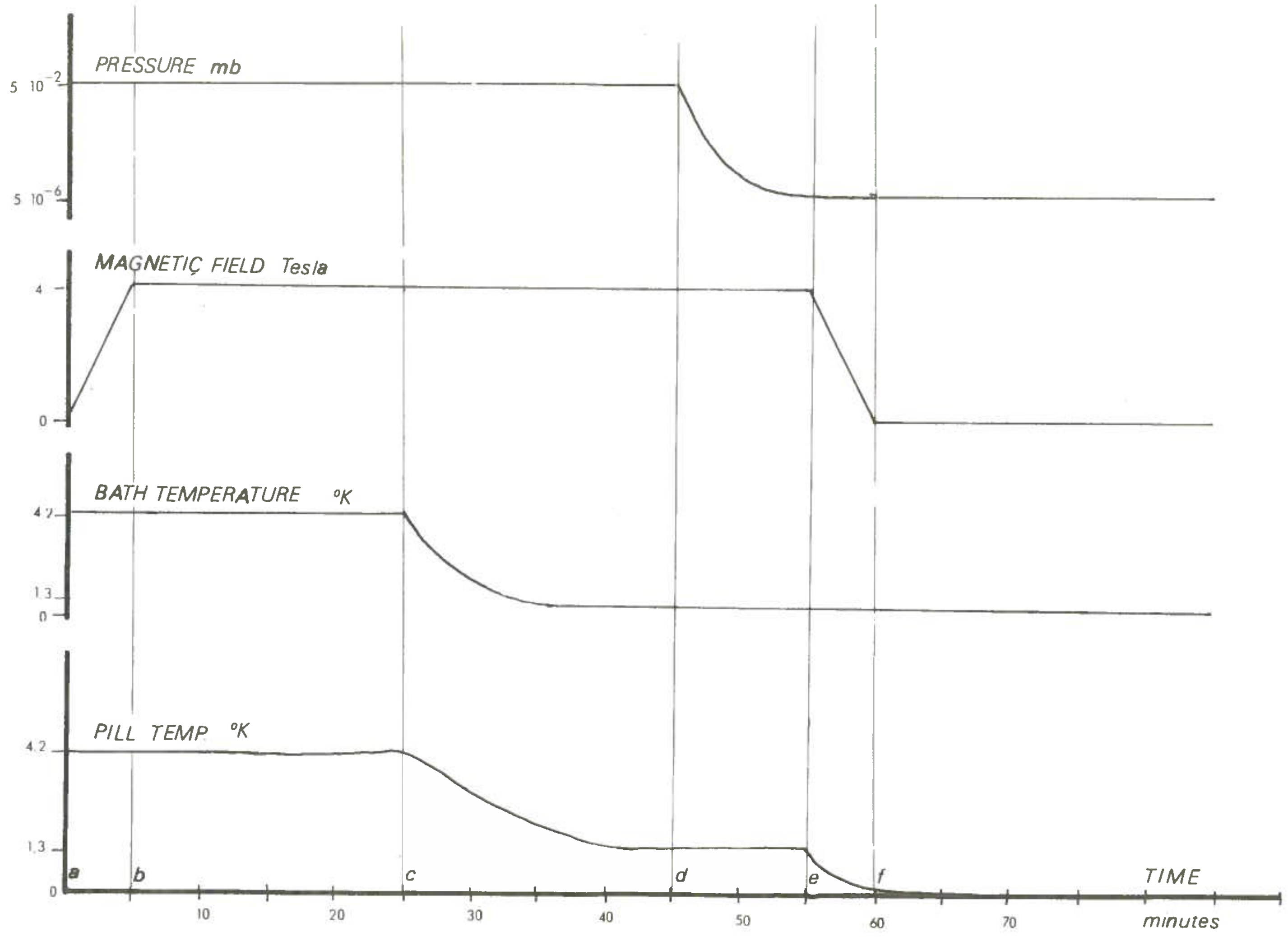

Fig.4 Time diagram of operation. (a)Start conditions.(b)The magnetic field is increased until 4 Tesla, the superconducting switch is ON and the power bars are extracted.(c)The He vapour pressure bath is reduced and the salt temperature decreases.(d)The cold salt is thermically disconnected from He bath by pumping the exchange gas.(f)Final conditions.

The vacuum tight low temperature insert of the cryostat is immersed in a pumped He4 bath in a commercial stainless steel dewar (Vaugam & Cameron,Ø15cm). Inside this insert a cylindrical salt sample (D=2cm,h=5cm) is hanged coaxially with a stainless steel tube terminating in the experimental chambre. The tube is surrounded by the superconducting solenoid immersed in the liquid Helium.

The salt, supported with a pirex pipe of small thermal conductivity is placed in the region where the magnetic field is most uniform. Copper wires furnish a tight thermal coupling between salt and the device under test in the experimental chambre. The internal pressure p of the exchange gas is controlled by the inlet valve v1 and the outlet valve v2. During the isothermal magnetization p is about 5.10**-2mbar. For the adiabathic demagnetization and the experimental phase p is reduced below 5.10**-6mbar by means of a diffusion pump and a liquid nitrogen trap.

2. - THE MAGNET. The main features of the superconducting magnet which is attached to the low temperature insert, are reported in table n 1. The current in the magnet (40 amps for 4 T field) is controlled by a commercial Oxford superconducting magnet power supply.

TABLE 1.

leads NbTi wire (diam.=.03). 361 Filaments in copper matrix
copper : s/c = 1.25 : 1
D.int. = 35mm
d.ext. = 49mm
Bmax = 4.15 Tesla Inductance 0.6 Henry
Uniformity 1% in D=20mm sphere

It is evident from the timing diagram that the magnetic field has to be switched on for a rather long time in order to recover the starting temperature after rising the field. To reduce helium consumption the magnet is thus operated in persistent mode and the power bars are disconnected and extracted from the liquid helium bath during constant field operation. These 8 mm diameter brass conductors are electrically isolated with paint and can vacuum tight slide or rotate in their upper flange seats. Coupling to the magnet is obtained with a screw connection to thinner electrical leads.

Operation in persistent mode is made possible by the superconducting switch in parallel to the solenoid coil. The quality of the switch is not critical; the 2-3 ohm resistance in the open condition do not impose severe limits to the current ramp slope and our home-made device is quite satisfactory. It was realized connecting the two superconducting leads of the main coil through a 80 cm superconducting cable made with a high resistivity cupro-nickel matrix, in order to have a few ohm resistance in the normal state. Each lead is tin soldered to the superconducting cable for a length of 10cm. The rest of the cable (60 cm) is coiled on a cylindrical support and wrapped with a manganina heater. Both coils are then inserted in a 5 cm long stainless steel tube filled with silastic to maintain a faible termal coupling to the helium bath. The 1/4 watt heater can force transition to the normal state in a fraction of a second. Superconducting state is quickly recovered when the heater is switched off.

3. - THE SALT SAMPLE. The dimension of salt sample is limited by the magnetic field uniformity and by the need to avoid thermal contact with the walls. A cylindrical sample of 23mm diameter and 50mm length is usually employed; no improvement in minimum temperature reached was observed for samples of reduced length.

Best results were obtained by pressing at 1ton/cm**2 in a suitable stainless steel cylinder a fifty-fifty mixture of salt cristals and tensol glue, added, to increase thermal conductance, with a 5% in weight of thin isolated copper wires ,few cm long. The excess glue is expelled from the cylinder.

Glue reacting with crystallization water realesed under pressure must be avoided. Minimum pressure compatible to obtain a compact pill

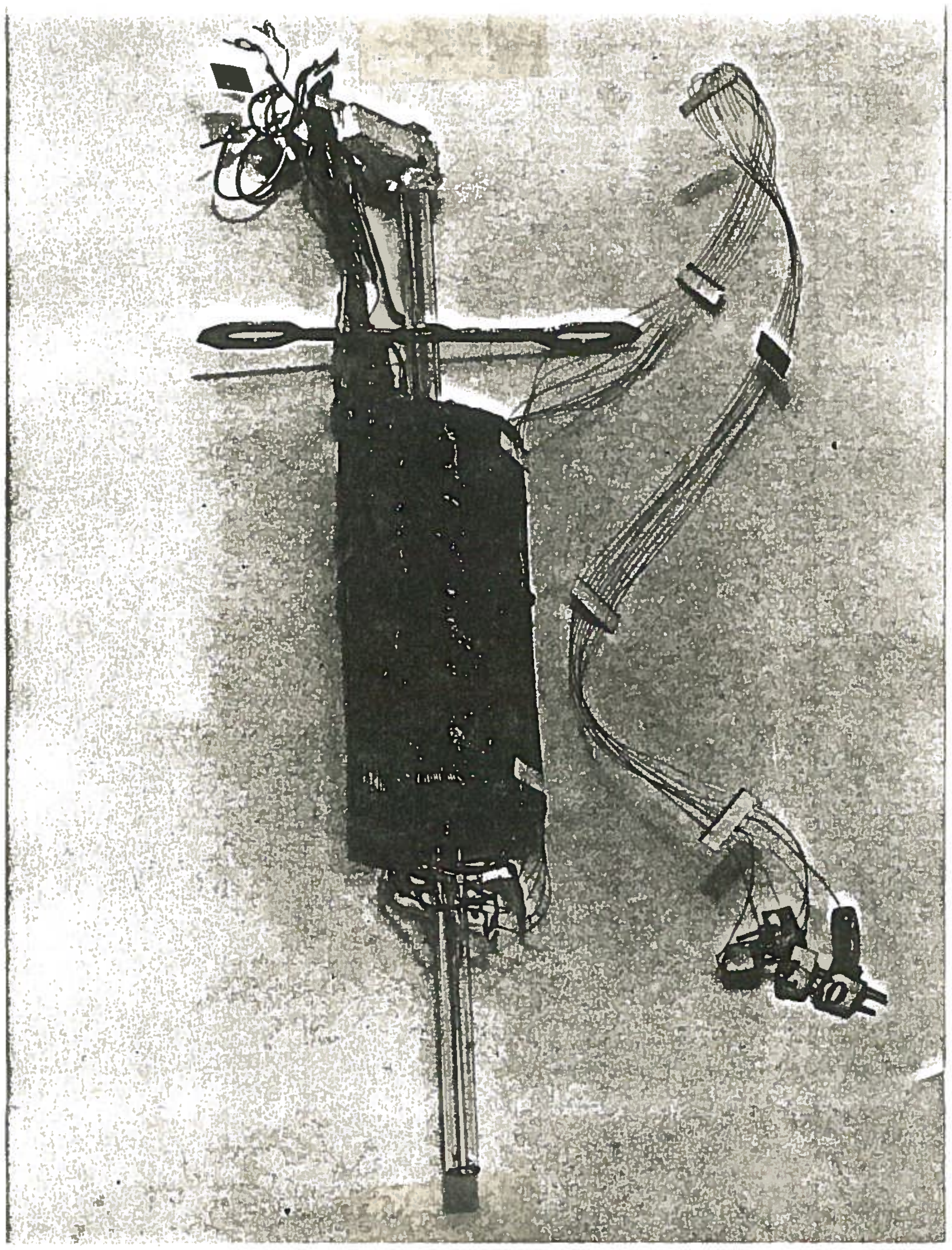

Fig.5 The salt pill after twenty thermal cycles.

should be used to reduce water loss which induces a rise of the magnetic ordering temperature and hence degrades the minimum temperature that can be reached. For this reason, during cryostat operations, pressure should never be reduced until the sample temperature is well under 0 C.

Due to the radial instability of a paramagnetic cylinder in an axial magnetic field, a strong support is needed to hold the sample in place . We use a 4mm diameter pirex tube inserted in an axial hole drilled through the salt and supported by vetronite spacers. Inside the pirex tube10 bulky isolated copper wires (D=.4mm) run. One end of these wires is tightly thermally coupled to the salt sample surface by wounding and gluing with

G.E.7031[(6)] varnish and is electrically connected to the external world with superconducting NbTi wires in brass matrix which are in thermal contact with the helium bath. The opposite end, terminating in the experimental chambre provides thermal and electrical connections to the device under test. Care was taken to minimize the effect of eddy currents by using aperiodic windings.

4. - TEMPERATURE MONITORING AND CONTROL. Calibrated carbon resistors(7) are used for temperature monitoring (8). They are usually placed on the salt sample and near the device under test on the heat sink made by the bulky copper wires.

A RuO thick film resistor connected to an A.C. measuring bridge is the sensitive element of the temperature control system; an 80 ohm manganina heater applied to the copper heat sink is the feedback element .

Thermal coupling between salt sample and copper sink is loose enough to allow temperature control within 1mk in the range 100mk-750mk with the salt sample below 100mk.

5. - CONCLUSIONS. The instrument has been in operation in our laboratory for more than half an year, mainly to select semiconductor materials with resistivity dependent on temperature in this range.

We were so enabled to realize bolometers, operating at 100mK, with sensitivity of 10**7 volt/watt.

Presently the instrument is employed to study the behaviour of the bolometers as X-ray detectors.

During this period no inconvenience was observed. After a few test runs no failure in reaching temperatures lower than 100mK was registered. The salt sample behaviour is stable over many termal cycles.

Temperature stability of 1mK around 100mk is achieved. The ultimate limits that can be obtained with the feedback control with improved sensors should be investigated.

The practical use of the instrument is so satisfactory that we are considering the construction of a second unit with increased experimental space.

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