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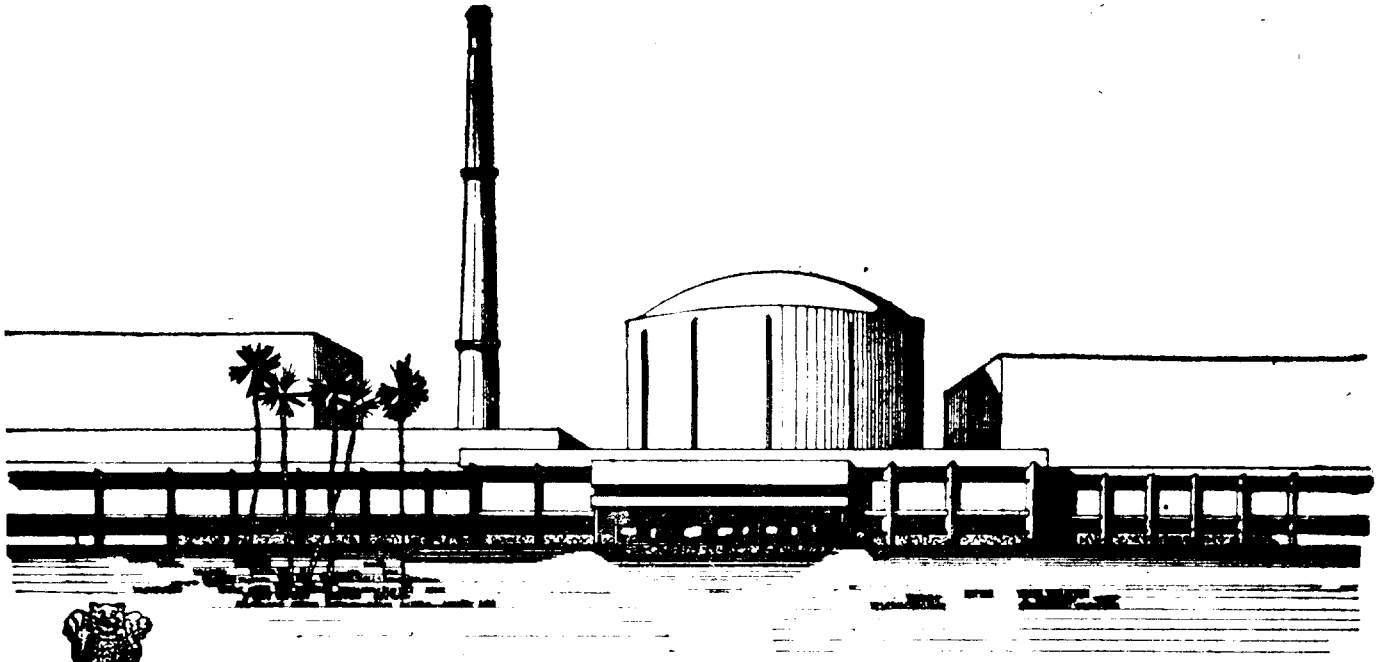

GOVERNMENT OF INDIA. DEPARTMENT OF ATOMIC ENERGY

INDIRA GANDHI CENTRE FOR ATOMIC RESEARCH KALPAKKAM

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DEVELOPMENT OF REVITALISATION TECHNIQUE FOR IMPAIRED LITHIUM DOPED GERMANIUM DETECTOR

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INTRODUCTION

Semiconductor detectors play very significant role in photon detection and are important tools in the field of Gamma Spectroscopy. Lithium doped Germanium detectors belong to this category. Initially hyper pure crystals of Germanium were scarcely available, and hence Lithium doping was accomplished. A Ge(Li) detector system is a germanium diode having a P-I-N structure mounted in a cryostat consisting of a vacuum chamber thermally coupled to a liquid nitrogen heat sink.

The "raw" material for Ge(Li) detectors is p-type grown single crystal Germanium having very specialized characteristics to enhance charge collection and lithium mobility. Under conditions of reverse bias at temperatures in the range of 25 to 40° C, the n-type lithium ions will migrate slowly towards the centre of the detector element. These n-type impurity atoms take interstitial positions within the crystal lattice and compensate for the original p-type impurity atoms on a one - for - one basis with the result that the net impurity concentration within the drifted region is of intrinsic proportions.

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Since the mobility of lithium ions in germanium must be very high in order for them to migrate the 20 mm or more that is required, it follows that they do not stay in place at ordinary temperatures once the drifting process is complete. In fact, serious lithium precipitation occurs within hours at room temperature and thus it is important that Ge(Li) detectors be kept at or near liquid nitrogen temperature at all times.

LIthium mobility is by no means the only reason a Ge(Li) detector must be cooled. At elevated temperatures, the thermal generation of charge carriers and the attendant leakage current is so great that the noise from the detector is overwhelming. The remarkable energy resolution of a Ge(Li) detector can only be attained with detector temperatures at or near that of liquid nitrogen.

CRYOSTAT DESCRIPTION

A cryostat consists of a vacuum chamber which houses the detector element plus a dewar(double wall vacuum - insulated vessel) for the liquid nitrogen cryogen. In some cases, the detector chamber and dewar share a common vacuum. These are called "integral" cryostat. "Dipstick" cryostat have a detector vacuum chamber with a dipstick-like cold finger which is inserted into the neck of a dewar. The detector element is held in place by a holder, which is electrically isolated but thermally connected to a copper cold finger. The cold finger transfers heat from the detector assembly to the liquid nitrogen reservoir. The detector holder is held in place by an anti-microphonic stabiliser. The detector holder as well as the outer vacuum jacket or "end- cap" are made as thin as

possible to avoid attenuation of low energy gamma rays. The holder is generally made of aluminum and is typically 0.5 to 1 mm thick.

Cryostats are vacuum baked at high temperatures to reduce out-gassing. Vacuum maintenance is accomplished through the use of molecular Sieves located in the tailstock of the cryostat. The amount of sieve material is adequate to maintain vacuum for more than 10 years under normal use.

VACUUM SEAL: Canberra dipstick cryostats have one main seal where the end-cap joins the cryostat body. In the typical cryo-stat this is an ultra high vacuum bakeable metal seal made by deformation of the end-cap material between annular pinch ridges in the fixed and floating flanges. A second seal is required for cryostat evacuation.

PROBLEMS EXHIBITED BY RDL Ge(LI):

There are very limited number of Ge(Li) detector failure modes, the most common being warm up and cryostat vacuum loss.. The number of things which contribute to loss of resolution are limitless, however, this is where careful diagnosis is more important.

RDL Ge(Li) detector started showing condensation at its surface which was indicative of vacuum loss in the cryostat. The detector was carried to TIFR(Bombay) for repair, and the restoration of vacuum was accomplished but it could not remain permanent. In 1982 again the problem cropped up. This time condensation was

severe followed by extremely high consumption of liquid nitrogen, even reaching a level of 30 litres per day(in normal condition about ONE litre per day). This time TIFR refused to take up repair work. We were left with only two options, either discard the Ge(Li) which was no doubt ten years old, or, to take up repair works at RDL. We chose the later and the planing was made.

REVITALISATION TECHNIQUE:

The repair works proceeded in the following steps:

1. To begin with the urgent need was to prevent any drifting and precipitation of lithium and hence round the clock shift was arranged to pour liquid nitrogen in the Dewar. The detector was wrapped in asbestos cloth to minimise reduction of cooling to the detector.

2. Diagnosis started for finding out the real cause of tremendous vacuum loss in the cryostat.

3. After due investigation it was found that the vacuum loss was not due to any damage of high vacuum seal but it was due to corrosion in the electrical feedthrough pins , connecting preamplifier (Fig 1).

4. The remedial steps were accomplished in two stages:

STAGE 1 :

Removal of corroded pins and sealing of the leakage with high vacuum sealant was carried out. The remaining pins were cleaned with fine Emery cloth and the dust was blown off,

followed by washing of pins with a mixture of methyl alcohol and hexane. Finally, hot air gun was used to dry the electrical feedthrough. The pins were given black wax trace coating to prevent further corrosion,and thereby avoiding the recurrence of the problem.

STAGE 2 :

Setting up of a system which can be used for reactivating molecular sieves, evacuating cryostat and completing thermal recycling of the detector (Fig 2). In this stage the vacuum seal was broken and a vacuum valve was attached instead. For trapping ions coming out of molecular sieves, a liquid nitrogen large vacuum flask was fabricated at RDL. The cryostat was attached to the system after removing from Dewar, and, then high vacuum was applied using oil diffusion pump. The pressure of an order of 10-5 torr was maintained which was being constantly measured using Penning gauze.

The cold finger (copper) dipped in liquid nitrogen plays the role of cooling the detector through conduction principle. Before giving heat treatment to the cryostat, the cold finger was put inside a thermocoal box containing liquid nitrogen and plugging any voids with fine asbestos powder and cloth. This step is prerequisite to preventing detector getting warmed up. Further, the asbestos cloth was wrapped spirally around the cryostat and the hot air was blown through the inlets of the spiral wrapping. After 30 minutes thermal cycle operation the adsorbed ions in the molecular sieves started coming out which was evident from the increase of the pressure (10-2 torr). The released ions were

trapped in the liquid nitrogen trap incorporated in the system and the thermal recycling continued until the pressure attained a level of 10-5 torr.It is worth remembering that during these operations the liquid nitrogen was, of and on,poured into the thermocoal box to ensure the availability of liquid nitrogen in the box. After completing the above cycle the thermocoal box was removed and the cryostat was dipped in the liquid nitrogen Dewar and system was allowed to stabilise over 48 hours before testing the performance. The above cycle of operations was repeated over a couple of days unless the resolution improves.

After testing resolution of Ge(Li) was fond to very much satisfactory and the consumption of liquid nitrogen was also restored to the normal conditions (about ONE litre a day). The Ge(Li) kept on performing as per the specifications till 1987. However, from 1988 deterioration of performance started due to its aging and the detector required redoping for regaining the performance which could not be possible owing to extremely high cost and lack of facility. The detector was, therefore, discarded in 1989.

Presently, only hyper pure germanium detectors are used for their simplicity and less maintenance as these need liquid nitrogen during operations whereas Ge(Li) needs all the time. The resolution of HPGES may also be improved using above technique.

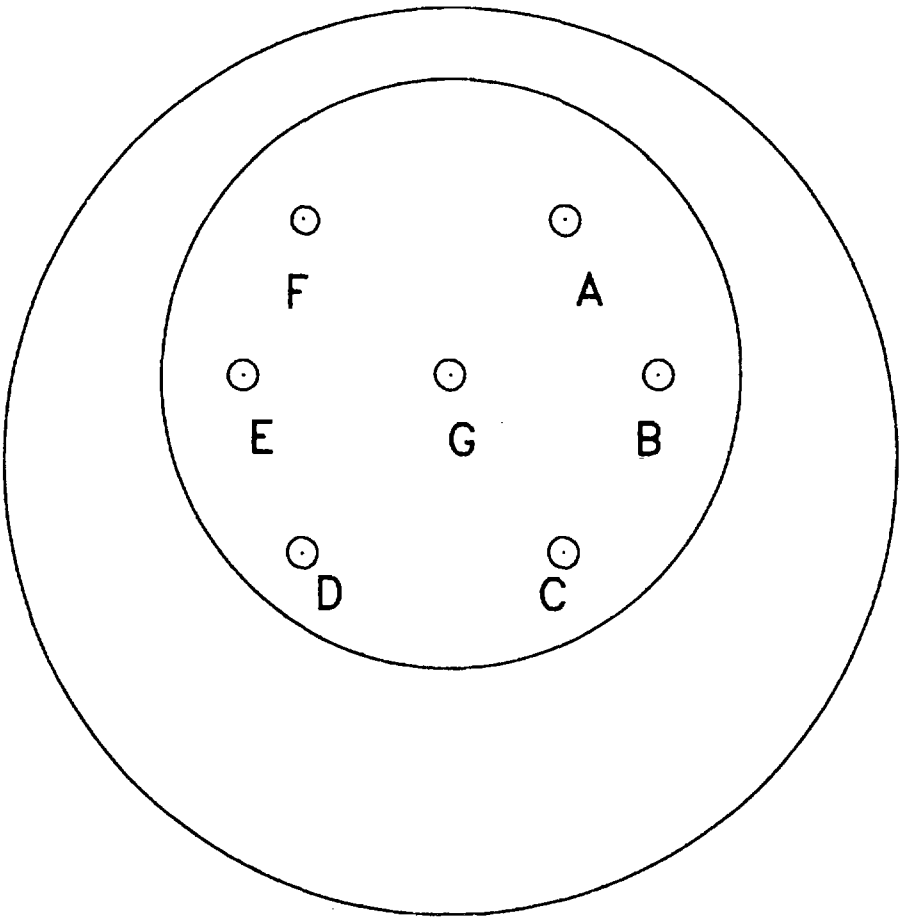

FIG 1

D.C. COUPLED : B OR E -- SIGNAL
G,B OR E -- BIAS

ELECTRICAL FEEDTHROUGH

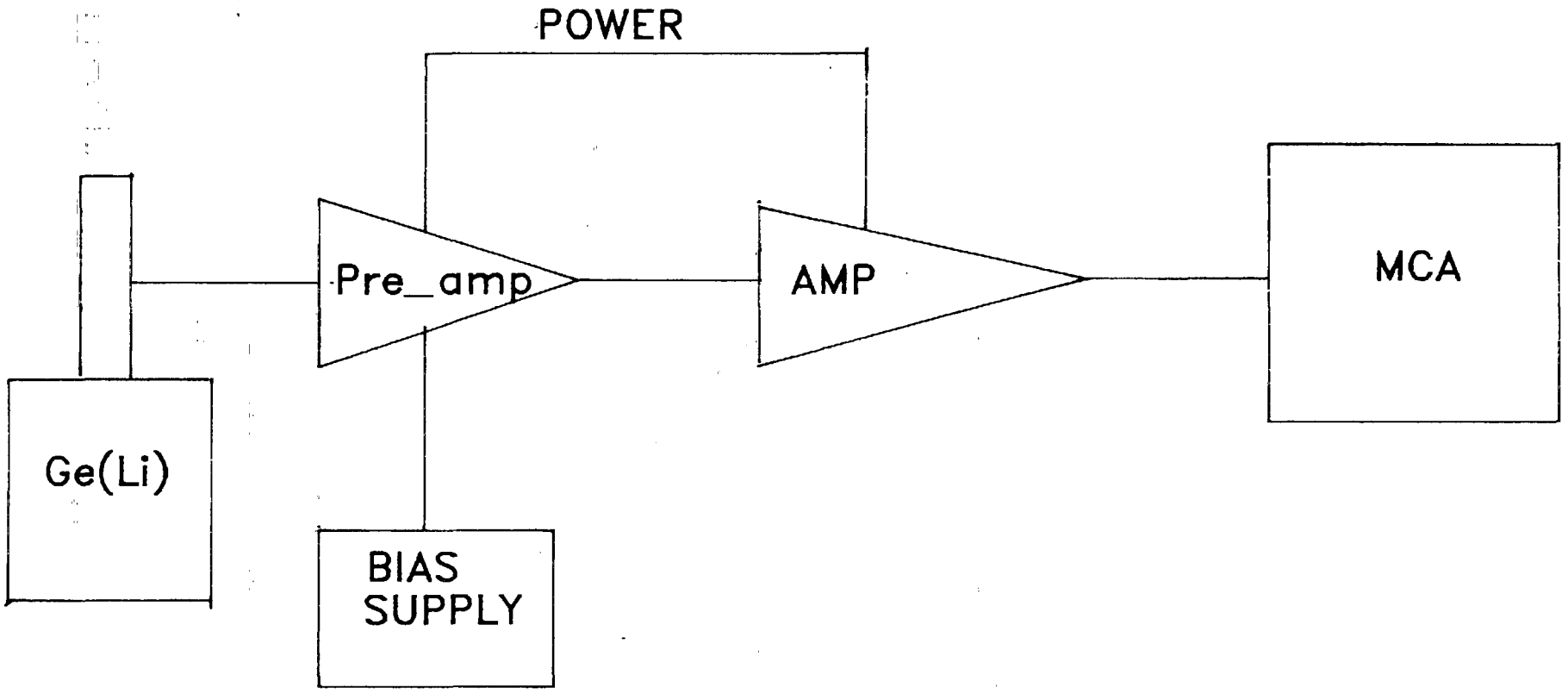

Fig 2

EQUIPMENT CONNECTION

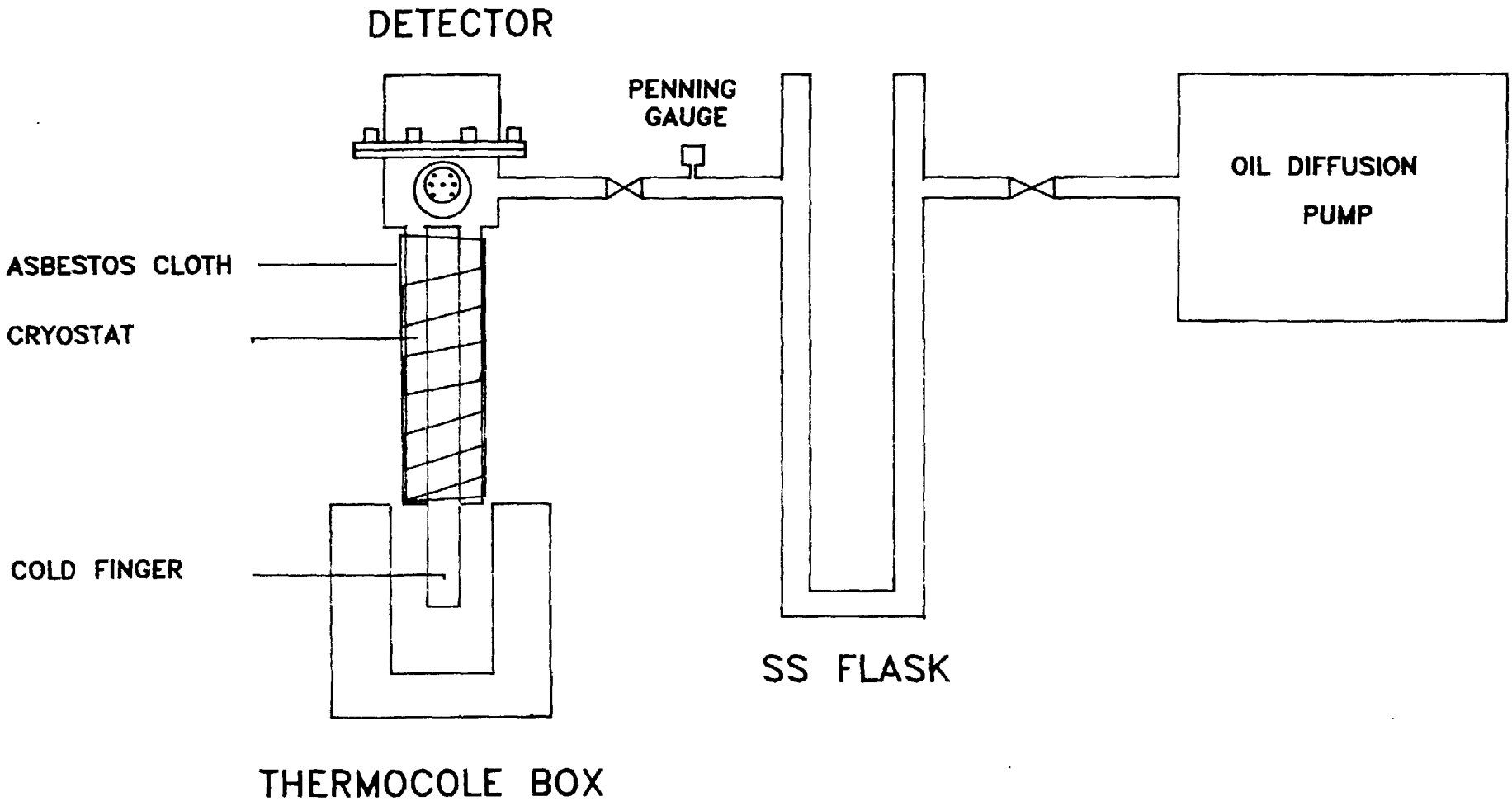

FIG 3

REVITALISATION SYSTEM