



Australian Radiation Laboratory

The Design of a Calorimetric Standard
of Ionising Radiation Absorbed Dose

by

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ABSTRACT

The design of a calorimetric working standard of ionising radiation absorbed dose is discussed. The background to the project is indicated, and a brief history of the appropriate quantities and units of measurement is given. Detailed design considerations follow a summary of the relevant literature. The methods to be used to relate results to national standards of measurement are indicated, including the need for various correction factors. A status report is given on the construction and testing program, including the solutions to several technical problems that have been encountered to date.

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i. INTRODUCTION

As described by the National Measurement Laboratory (NML 1977), a primary standard is a device or measuring system, operated by a single laboratory in each country, defining the value of a physical quantity for use in that country. The standards maintained by different countries for a particular physical quantity are occasionally compared, to ensure uniformity throughout the world. Secondary or working standards for a particular physical quantity are compared with the national primary standard regularly and are used by secondary standardising laboratories to provide a calibration service for instruments used throughout the country.

The Australian Radiation Laboratory (ARL) is authorised, by the Commonwealth Scientific and Industrial Research Organisation (CSIRO), under the Weights and Measures (National Standards) Act 1960, "... to maintain working standards of absorbed dose of ionising radiation" (CSIRO 1977). The Australian Atomic Energy Commission (AAEC) is authorised under the Act to maintain the Australian Primary Standard of absorbed dose.

These standards are realised using equipment which is calibrated against national standards of basic physical quantities, such as mass, length, time and electric current, maintained by the National Measurement Laboratory (NML) of the CSIRO (now called the Applied Physics Division of the CSIRO).

Ionising radiation standards are maintained as described above, in order to ensure accuracy and uniformity of measurement throughout Australia and the world, in the fields of radiation protection and radiotherapy. As knowledge advances, scientific results in all fields tend to be reported and used with greater precision. This increases the need for precise and appropriate standards of measurement, to allow meaningful comparisons of such results.

1.1 History of Radiation Dose Units

Ionising radiations have been studied since X-rays were discovered by Roentgen in 1895. Eisenlohr (1978) points out that as early as 1913, an absorbed dose quantity was proposed by Christen, as being most directly related to the effects of ionising radiations on matter. The technology of the time was not equal to measurement of this quantity however, and in 1928,

the Stockholm Radiological Congress defined a quantity based on the charge liberated from a known mass of air by a beam of X or γ radiation. They called the unit for this quantity the roentgen, which now has the symbol R. The roentgen is relatively easy to measure for photon energies between a few keV and a few MeV, but outside these limits it becomes extremely difficult to fulfil the requirements of the definition, and modern radiotherapy machines can produce X-rays of up to 50 MeV. A recent report by Attix (1979) indicates a possible method of extending the use of the roentgen to higher X ray energies, but this is only a tentative proposal at present. Furthermore, the roentgen is defined explicitly in terms of X and γ radiations only (Cember 1969, p.151).

In 1953, the International Commission on Radiological Units and Measurements (ICRU) defined the rad as the unit of the quantity Radiation Absorbed Dose, 1 rad being defined as 100 erg per gram (Eisenlohr 1978). This quantity applied to any type of radiation, not just X or γ rays.

The ICRU redefined the roentgen in 1962 as exactly 2.58×10^{-4} coulomb per kilogram, and at the same time gave the quantity for which the roentgen is the unit the name "exposure" (Cember 1969, p. 151). A new S.I. unit was introduced for the radiation absorbed dose by the ICRU in 1975. The gray (symbol Gy) was defined as one joule per kilogram (100 rad). (Wyckoff 1978).

The quantity radiation absorbed dose is now formally defined (ICRU 1980) as the quotient of $d\bar{\epsilon}$ by dm , where $d\bar{\epsilon}$ is the mean energy imparted by the radiation to the matter (of mass dm) in an infinitesimal volume element, at the point of interest. This may also be expressed as the limiting value (for small m) of the mean specific energy imparted, \bar{z} ;

$$D = \frac{d\bar{\epsilon}}{dm} = \lim_{m \rightarrow 0} \bar{z}$$

where $z = \frac{\epsilon}{m}$ = energy imparted per unit mass. (specific energy imparted)

The quantity z is a stochastic quantity that applies only to a finite volume. Owing to the discrete nature of radiation interactions with matter, the precise value of z cannot be predicted for any given set of irradiation conditions. The absorbed dose however, can be predicted, as it is the limiting value of the mean of z . As m becomes very small, z can become either very small or zero (no energy deposited in the volume element during

irradiation) or very large (total absorption of one or more photons etc. in the volume element during irradiation). D however is taken as the limit of z for larger m as m tends to zero (Attix and Roesch 1968).

1.2 Uncertainty in Radiotherapy Dosimetry

Radiotherapy dosimetry is carried out in hospitals and clinics throughout the world. It is necessary to control the dose delivered to the patient to an accuracy of a few percent. The U.S. Committee for Radiation Therapy Studies has recommended that the absorbed dose to a measuring instrument in a phantom should be known with an uncertainty not exceeding three percent, and that the absorbed dose delivered to the patient should be within five percent of the prescribed dose (Lanzl 1978). A five percent change in the absorbed dose to the patient has been reported to have a marked clinical effect in certain radiotherapy procedures (Eisenlohr 1978).

The present method of controlling radiotherapy dose is to use exposure-calibrated instruments with calculated conversion factors, to obtain the absorbed dose (Massey 1970). The values of the factors for high energy X-rays (which are now universally used in radiotherapy) are still subject to review in the literature, and have associated with them an uncertainty of a few percent. Thus there may be up to a 5 percent uncertainty in the determination of the absorbed dose to a water phantom. Further uncertainty is introduced in calculating from this the absorbed dose to the patient.

2. ABSORBED DOSE STANDARD

A graphite microcalorimeter is under development at ARL to provide a more direct method of calibrating dosimeters, eventually in terms of the absorbed dose to water. The national standard of radiation absorbed dose is realised by the AAEC using a similar graphite microcalorimeter. Microcalorimetry is a very difficult measurement technique, requiring complex equipment close to the limit of technology. Meticulous precautions must be taken, to achieve a reasonable signal to noise ratio, while maintaining direct traceability of the measurements to national standards of basic physical quantities. Such complex equipment, including highly delicate and sensitive detection and control devices, is prone to failure, requiring long repair times. This leads to a need for good stable working standards calibrated against the calorimetric primary standard. Both AAEC and ARL now have ionisation chamber instruments,

calibrated for radiation absorbed dose, for cobalt-60 γ radiation (1.17 + 1.33 MeV) against the existing national primary standard calorimeter. The ARL calorimeter will become a further working standard, of primary standard quality. This will not depend on frequent comparison with the national standard for accurate measurements of the absorbed dose. In particular, it is hoped that the calorimeter will be useful for higher energy X-rays, as produced by typical medical accelerators, to provide direct measurement of the absorbed dose in calibrating dosimeters. No such direct calibration facility is available at present in Australia. Linear accelerators generally produce radiation in very short, intense pulses, which can lead to ion recombination losses in ionisation chamber detectors. A calorimeter would not be subject to this problem.

3. LITERATURE SURVEY

Preliminary results of a literature survey indicate that practical absorbed dose calorimeters have been constructed, mainly by scientists working in leading national standards laboratories, since 1958. Most have used carbon as the absorbing element, in the form of graphite, because of its approximate tissue-equivalence (atomic number 6, specific gravity 2.7) coupled with an insignificant heat defect. The heat defect is the difference between the amount of energy deposited in the absorber by the radiation, and the amount appearing as heat. A heat defect may arise due to chemical reactions. Carbon calorimeters have been described by Reid and Johns (1961), Petree and Ward (1962), Bewley (1963), Geisselsoder et al. (1963), Bradshaw (1965), Henry (1972), Hohlfeld (1973), Sabel et al. (1973), Guiho et al. (1974), Guiho and Simoen (1975), NBS (1975), NPL (1975), PTB (1975), Zsdansky (1975), Urquhart et al. (1978) and Sundara Rao and Naik (1980). Calorimeters using aluminum absorbers have been described by Reid and Johns (1961), Petree and Ward (1962), Schleiger and Goldstein (1964) and Urquhart et al. (1974). Tissue-equivalent plastic has also been used, as in Milvy et al. (1958), Genna et al. (1963), Sabel et al. (1973), Greene et al. (1975) and McDonald et al. (1976). A recent report (Domen, 1980) describes the direct measurement of the temperature rise of water in a calorimetric measurement of radiation absorbed dose.

Typically, construction is aimed at thermally isolating the absorber as well as possible from ambient conditions, in order to measure accurately the very small temperature rise induced in the absorber by the radiation beam. The temperature rise is typically a few millikelvin. The absorber is usually suspended by small cross-section supports of insulating material (points or strings) inside one or more temperature controlled (isothermal, isoperibol* or adiabatic) jackets in an evacuated vessel.

Radiation types used have ranged from X-rays and cobalt-60 γ rays to electrons and neutrons. Absorbed dose rates have ranged from less than 10 mGy/min to over 80 Gy/min.

4. MICROCALORIMETER DESIGN

Drawing on the literature, an experimental graphite microcalorimeter has been designed, and construction is nearing completion (see figure 1). A small graphite disc A, 3mm thick by 25mm diameter is held suspended within three graphite jackets B, C and D, a steel vacuum vessel E and an insulated plastic box G. Thin aluminised mylar** windows F at the ends of the cylindrical vacuum vessel allow the radiation beam to enter and leave the calorimeter, without encountering any significant amount of non-graphite material which would alter the characteristics of the beam. The graphite parts are suspended within each other by thin nylon threads, secured at the outer edge only of each jacket. The threads pass through clearance holes in each jacket, to cross the small gap to the next component within, see figure 2. Other small passages are left through each jacket to allow for the extraction of air during pumping down, and for electrical connections. As the calorimeter is not a completely solid block of carbon, correction factors (hopefully small) will have to be evaluated to allow for the presence of foreign materials. Non-graphite materials in and around the absorber include wires, thermistors (temperature-sensitive resistors used to detect the temperature rise), heaters, vacuum gaps, reflective coatings, adhesives, etc.

* Isoperibol means constant temperature surroundings.

** Mylar is polyethylene terephthalate film used extensively in radiation and vacuum work.

The depth of the absorber below the surface of the calorimeter's outer-most graphite jacket or mantle will be approximately 5 g/cm^2 , which is the recommended measurement depth for the dosimetry of X or γ radiation of energy between 150 keV and 10 MeV (ICRU, 1973).

Graphite and water phantoms will be constructed, to allow the measured absorbed dose in the calorimeter to be transferred to water, using an ionisation chamber. This will need to be done extremely carefully, as it may prove to be the largest source of uncertainty in determining the absorbed dose to water. The graphite phantom will also be used to find some of the necessary correction factors. These corrections include allowances for the differences in attenuation of the radiation beam for different detectors to be calibrated, and the effect of some of the necessary gaps inside the calorimeter.

4.1 Absorber Power Input and Temperature Rise

The only suitable radiation source available at present is a 20 TBq (550 Ci) cobalt-60 teletherapy source, which delivers about 100 mGy/min at 1 meter. As $1 \text{ Gy} = 1 \text{ J/kg}$, this corresponds to a specific power input of $1.7 \text{ } \mu\text{W/g}$. The absorber mass is approximately 2.5 g so that the expected power input is $4.2 \text{ } \mu\text{W}$.

The specific heat of the graphite used is about $730 \text{ Jkg}^{-1}\text{K}^{-1}$, so that in a typical exposure of 1000 seconds duration, the temperature of the absorber is expected to rise about 2.3 mK. Note that the temperature rise is independent of the actual mass of the absorber, depending only on the radiation field strength (and uniformity), and the specific heat of the absorber:

$$D = \frac{d \epsilon}{d m} = c \Delta T$$

4.2 Irradiation Conditions

The calorimeter will be placed in a radiation beam large enough to completely irradiate both the absorber and the innermost jacket. The second jacket will be temperature-controlled to follow the temperature of the first

jacket. This will ensure that no significant heat transfer occurs between the two jackets, or between the first jacket and the absorber. The resistance change of a thermistor (in thermal contact with the absorber) will be monitored during this period, by recording the drift from balance of a wheatstone bridge, having the thermistor as one arm. Drift in the bridge off-null voltage before and after the exposure will be allowed for, probably as described by Gunn (1971).

4.3 Calibration Procedure

After irradiation, the energy input corresponding to the observed change in the bridge null voltage will be found, by passing a measured current through a heater in the absorber. This will be adjusted to cause the thermistor resistance to follow (as closely as possible) the change that was observed during irradiation. The duration of heating will be measured using a quartz crystal controlled timer. The voltage developed by the current in the heater during calibration will be measured by means of separate potential leads. The current will be found by measuring the voltage developed across a standard resistor.

The heating current should develop about 60 mV across both the 1000 ohm heater and the 1000 ohm standard resistor, at the expected power input of about 4 μ W. It is expected that initially, only a few voltage and current measurements will be used to calculate the electrical input power during the calibration heating. Later it may become desirable to take many samples of the instantaneous voltage and current values, using a computer to calculate a more precise value of the total energy input. The DVM, timer, standard resistor and absorber mass will all be made traceable to national standards of measurement.

4.4 Absorber Heater Details

The absorber heater will comprise a layer of graphite-loaded epoxy resin sandwiched between layers of plain insulating epoxy as described by Hohlfield (1973). This composite will form the joint between the two halves of the graphite absorber disc. The resistance of the heater will be about 1000 ohm. In order to achieve the most uniform heating possible, the current leads will

extend across the heater disc in two chords as shown in figure 3. Potential leads are shown, connected one at either end of the gap between the absorber and the first jacket. The intention of this method of connection is to include in the measurement half the voltage drop which occurs in the short lengths of the current leads crossing the gap to the first jacket, thus including half the heat generated in these parts of the wires due to their own resistance. This accords with standard calorimetric practice (Martin 1972). The heater leads will be allowed 1mm of slack as they cross the 0.5mm gaps between each jacket, to prevent breakage and to decrease heat conduction. This is discussed further in section 4.5.

Manganin wire was chosen for the current leads. This is a compromise between a reasonably low resistance and a fairly low thermal conductivity. A low resistance is achieved over the chord length crossing the heater disc (about 10 ohm or one percent of the heater resistance between the leads) to provide a reasonably uniform current density throughout the heater. A low thermal conductivity is required to minimise heat transfer along the wires crossing the narrow gap to the first jacket. The resistance of the wires crossing the gap is expected to be about 0.5 ohm or 0.05 percent of the heater resistance, thus heat generated in the wires is expected to contribute about 0.025 percent of the energy input to the absorber during calibration.

In the case of the heater current leads, a short length of wire crossing the gap is deemed preferable to a larger span of wire having a lower electrical resistance and a higher thermal conductivity. If (for instance) copper wire were used, it would need to be extremely thin, and hence fragile, in order to have a sufficiently low thermal conductivity.

Several alternative routes for the heater current leads were considered. They could be taken with the other leads through a gap in the first jacket, passing through the second jacket (in thermal contact with it) to the vacuum vessel wall. The temperature control of the second jacket however, may be very much inferior to that of the first jacket. In this case, unacceptable heat transfer could occur, as these longer wires would necessarily be of lower electrical resistance and hence have higher thermal conductivity than the manganin wire selected.

The physical properties of the various types of wire considered for these necessary electrical connections are shown in table 1.

It would be possible to use a coil of nichrome or evanohm wire as a heating element for the absorber. A half-meter length of 0.001" diameter nichrome wire would have a resistance of about 1000 ohm. Such a coil would have a wire spacing of about 1mm over the entire area of the absorber disc. This would probably be sufficiently uniform to accurately simulate the radiation heating. The mass of such a coil of wire would be about 2 mg or 0.1 percent of the total absorber mass. While this is not large, the mean atomic number (Z) of such wire is about 25, which is markedly different from that of carbon ($Z=6$), water ($Z=8$) or tissue ($Z=7.6$ - see Johns and Cunningham, 1969). The epoxy resin heater is therefore regarded as a better alternative, as the mean atomic number of the epoxy resin mixture used is about 6.

4.5 Heat Transfer Between Graphite Parts

Three mechanisms of heat transfer must be considered in order to achieve good thermal isolation of the absorber. Convection will be eliminated by evacuating the spaces between the various parts of the calorimeter until the mean free path of the remaining air molecules exceeds the gap sizes. It is expected that a vacuum of about 10 mPa (10^{-4} torr) will be required for this. Although some two stage rotary pumps can achieve this order of vacuum at the pump inlet port, it is expected that a diffusion pump will be required in order to achieve a sufficient vacuum inside the calorimeter, in a reasonable pump down time.

Radiative heat transfer could be reduced by polishing or coating the surfaces of the absorber and first jacket. Coating is to be avoided if possible as it would introduce foreign material, contaminating the pure graphite environment, and requiring a larger correction factor. Some success has been achieved in polishing the graphite surfaces with very fine emery paper. An experiment is planned to measure the reflectance of these polished surfaces for infra red radiation.

Both radiative and conductive heat transfer can be reduced by maintaining low temperature differentials between the various parts. Temperatures of the various parts are expected to be controlled to the levels shown in table 2.

Conductive heat transfer can be controlled by reducing physical contact between the various parts to the bare minimum. The suspension threads have already been mentioned. The only remaining physical contacts are the electrical wires. These have been chosen carefully as indicated in the previous section for low thermal conductivity, and routed so as to minimise the expected temperature differentials between the ends of the wires passing from one part of the calorimeter to another. Temperature control aims have been assigned arbitrarily to the various parts of the calorimeter. As the temperature change to be measured at the absorber is on the order of 1 mK, it seems reasonable to aim at controlling the temperature here to 1 μ K. Succeeding jackets could then be permitted to have temperature variations increasing by an order of magnitude for each jacket. If these aims are achieved, peak to peak excursions of the order shown in table 2 are expected. Thus temperature differences of this order may be anticipated between each part and the next inner part. This allows calculation of the maximum permitted rate of heat transfer, for selection of suitable wiring connections.

The absorber heater current leads have already been described. These leads will pass across the gap between the absorber and the first jacket with only 1mm free length, to minimise heat generation in this part of the wires. The leads will then be in thermal contact with the first jacket, which will be heated to follow the temperature of the absorber. It is estimated that if the temperature control aims for the various parts can be achieved (table 2), then the maximum heat transfer along the heater current leads between the absorber and the first jacket, due solely to temperature differences between the two parts, will be about 1 nW (table 1). The evanohm potential leads, and the three other pairs of evanohm leads from the thermistors on the absorber, will be taken through an evacuation gap in the first jacket to the second jacket, with which they will be in thermal contact (figure 4). This is to provide extra wire length, which together with the lower thermal conductivity of evanohm wire, is expected to keep the total heat transfer to or from the absorber by way of these wires down to about 1 nW, even allowing for the possibly poorer temperature control which may be achieved in the case of the second jacket. Thus the total random heat transfer from all sources to and from the absorber (this excludes the thermistor self heating due to the bridge current, see section 4.6) is expected to be less than 4 nW or 0.1 percent of the power input from radiation. This is deemed acceptable.

The absorber heater current leads will be in thermal contact with each jacket, to provide the possibility of operating the calorimeter with a known, constant heatsink of about $10 \mu\text{W}$ (slightly exceeding the expected radiation power input). The absorber could be maintained at a slightly elevated temperature relative to that of the first jacket, by applying power to the absorber heater. A temperature difference of about 0.5 deg C should be sufficient for this purpose. If 1 mm of slack is left in the heater current leads across each gap, then the first jacket could be held at a temperature of about 0.5°C above that of the second jacket, thus providing about a $10 \mu\text{W}$ heatsink to the second jacket. Similarly, if the second jacket in turn were held at about 0.5°C above the temperature of the mantle, then the $10 \mu\text{W}$ heatsink would be passed along to the mantle. It is expected that the intrinsic heatsink from the mantle to the vacuum vessel will greatly exceed $10 \mu\text{W}$, so that this heat will easily be absorbed by the mantle temperature control circuit. Under irradiation, the decrease in power input required to maintain the absorber at a constant temperature would then indicate directly the power input from the radiation beam, and the system would be self calibrating. It may be possible to improve the sensitivity of this technique, which is inherently lower than that of the integrating method previously described, by chopping the radiation beam and using lock-in amplifiers. This may be investigated.

4.6 Heat Measurement Technique

A highly sensitive wheatstone bridge circuit will be used to detect the change in resistance of a thermistor on the absorber (figure 5). Before irradiation, R_D will be adjusted to bring $R_T + R_D$ to equal R , balancing the bridge, so that $V_1 = V_2$ and $I = E/2R$. After irradiation, R_T will have changed to $R_T + \Delta R$ (ΔR is negative, the temperature coefficient of the thermistor being about $-4 \text{ percent / deg C}$). Therefore the change in I is given approximately by

$$\Delta I = -\frac{\Delta R}{2R} \cdot \frac{E}{2R}$$

and the change in V_1 by

$$\Delta V_1 = R \Delta I = -\frac{\Delta R}{R} \cdot \frac{E}{4}$$

The temperature change is expected to be about 2 mK in a 15 minute exposure (see section 4.1),

so that

$$\begin{aligned}\frac{\Delta R}{R} &= -0.04 \times 0.002 \\ &= -80 \text{ ppm.}\end{aligned}$$

Thus the change in V_1 is expected to be (for $E = 0.5 \text{ V}$)

$$\begin{aligned}\Delta V_1 &= 80 \times 10^{-6} \times 0.125 \\ &= 10 \text{ } \mu\text{V.}\end{aligned}$$

Clearly this will not be an easy voltage change to measure and reproduce accurately. The thermistors chosen have a resistance of 100 kilohm at 20°C; at 30°C this reduces to about 60 kilohm. The bridge arms have $R = 100$ kilohm. A high resistance was chosen to allow the highest bridge voltage E , consistent with limiting the self heating of the thermistor. If the self heating rate is to be no more than 10 percent of the radiation input power, this requires E to be less than 0.5 V for 60 kilohm thermistors. This gives a self-heating of

$$\frac{E^2}{4R^2} \cdot R_T = \frac{0.25}{4 \times (100\text{k})^2} \times 60\text{k} = 0.4 \text{ } \mu\text{W}$$

This value of E was used in the above calculation of ΔV_1 . Note that the thermistor self-heating can be expected to be a constant heat source, so that a higher value can be tolerated than in the case of a probably variable energy input from neighbouring calorimeter parts. In order to measure the small voltage change ΔV_1 accurately, a special high resistance, carefully constructed wheatstone bridge has been designed and built (supplied by J L William Scientific Instruments), incorporating low thermal design and a driven guard facility. A highly sensitive, low noise null detector has been obtained with favourable noise performance at the high source impedance represented by the bridge.

4.7 Jacket Temperature Control

As previously described, it will be necessary to control the temperature of the first jacket during calibration to follow that of the absorber. This will be achieved using a differential wheatstone bridge with thermistors in adjacent arms, one thermistor being located on the absorber and the other on the first jacket. There will be two separate heaters in every jacket, one on each side of the absorber disc. These may be used either to even up the

radiation heating during exposure, by compensating for attenuation of the gamma ray beam, or to duplicate precisely the non-uniform heating by the beam during irradiation, by heating one part more rapidly than the other during calibration. Note that there may be only poor thermal contact between the two parts of each jacket, owing to the method of joining. This method of joining was selected to permit relatively easy disassembly of the calorimeter in the event of the failure of any of the many heaters, thermistors, wires, or welded joints. Thus should such a failure occur, in principle only the offending part, and probably also the absorber, should need to be replaced. This should save in both labour and material costs, and should also reduce the necessary repair time. Three thermistors will be installed on the absorber, although only two are needed for measurement and control purposes. This will provide one spare thermistor in case of a single failure.

Similarly, the second jacket may be controlled to follow the temperature rise of the first jacket during irradiation, and the outermost jacket (or mantle) may be made to follow the temperature rise of the second jacket. Alternatively, the outer jacket and mantle may each be held at some fixed mean temperature. Other temperature control facilities will exist for the vacuum vessel, the multi-way connector panel where cables may be connected to the various thermistors and heaters, and possibly for the measurement bridge itself. These last temperature control facilities may be needed to reduce thermal emfs, by maintaining the entire circuit at the same temperature (within a fraction of a degree celsius). For this reason also, most wiring in this part of the circuit will be done using plain copper cables and crimped plain copper lugs, or low thermal soldered joints to gold plated copper connectors.

5. EQUIPMENT AND CONSTRUCTION STATUS

Construction of the calorimeter is fairly well advanced. All the graphite parts have been made, the thermistors and special wires have been obtained, and a welding technique has been developed to connect the special, very fine wires to the equally fine, and very short thermistor leads. Most of the heaters have been made. The final absorber heater has yet to be made owing to a design change affecting the potential leads. Final assembly of the graphite parts is also waiting for the completion of an experiment designed to investigate the reflectance of the polished absorber discs, and on the

accurate weighing of the various parts of the absorber. Detailed consideration has been given to developing a step-by-step assembly procedure which will ensure that as much information is obtained as possible about the dimensions and mass of the various parts, and the gaps between them.

The vacuum vessel and its enclosing insulated box have been constructed, and tested for prolonged periods under vacuum. Heaters and thermistors which have been installed on both the vacuum vessel and the connector panel have been tested, and temperature control circuits involving wheatstone bridges and power amplifiers have been developed and tested. The temperature of the connector panel has been controlled using these circuits to ± 50 mK, and that of the vacuum vessel to ± 2 mK, exceeding the desired minimum degree of control (see table 2). Note that both the connector panel and the vacuum vessel were open to the room air during these tests. A recent report (Handschy 1980) gives details of a temperature controller capable of maintaining temperatures constant to within 100 μ K, with ambient temperature fluctuations of about 0.5 K. Thus it may be possible to improve on the temperature control achieved to date if this proves desirable. It was found that after an initial heating period, during which low gain was required to prevent oscillation (due to the fairly long time constant connecting the thermistor temperature and the heater temperature), the steady state power input required to maintain the vacuum vessel at a constant temperature above ambient was approximately 120 mW per degree celsius. Thus at a typical operating temperature of 10 degrees above ambient, the power required will be about 12 W. It took about 2 hours to reach the final steady temperature after switching on, from room temperature.

5.1 Welding Thermistor Leads

An unsuccessful attempt was made to use a high temperature (hydrogen flame) microwelder. Although satisfactory joints appeared to be made, most turned out later to have very high resistance and were extremely brittle. When examined under a microscope, these joints had a burnt appearance. It was concluded that the wire had been oxidised in the welding process.

It was felt that soldering was to be avoided if possible, in order to avoid any thermal emf problems. A capacitance discharge welder was therefore evaluated at Monash University, and was found to produce very satisfactory, low resistance joints in a highly repeatable manner.

There appears to be no commercial supplier of similar equipment at present, so a low power unit was designed and built, incorporating a cheap binocular microscope. Precision ball jointed electrode clips were also manufactured at this Laboratory. Provision is made for testing the resistance of the resulting joint immediately after welding, using the same electrode clips used to carry the welding current. A circuit diagram of the unit is shown in figure 6, and a schematic diagram of the welding clips in figure 7. Capacitors varying in steps of about a factor of 2 from 1 μF to 1000 μF can be connected in parallel, and charged to a metered voltage of up to 100 V by an external power supply. The supply is then disconnected and the capacitors are discharged rapidly through the twisted pair of wires. This generates a high temperature which melts the two wires to form a weld. A little experimentation is needed for each type of joint to be made. The capacitance can be increased to provide a longer current pulse, or the voltage can be increased to provide a higher peak current and hence a higher temperature. Results have been very satisfactory.

Another problem related to the welding of these fine wires was in removing the insulating varnish from the ends of the wires, without damaging them by scratching, stretching or bending, and thus weakening them.

After some initial difficulty, and extensive testing of different methods, it was discovered that concentrated sulphuric acid does an excellent job, without significantly attacking the wires.

5.2 Effect of Electrostatic Fields

Changing electrostatic fields in the vicinity of the high impedance bridge terminals (eg. from clothing) were found to induce large voltages into the bridge circuit. This has necessitated careful shielding of these terminals, involving the folding of sheet metal covers for the binding post bridge terminals, and even for the bridge battery compartment. It may be necessary to enclose the entire bridge in a shielded, temperature controlled box.

5.3 Electronics

Special interface units are being designed to allow switch selection of current or voltage measurements, using a single DVM, and to control the timer during calibration. Other interface units are being designed to control some

of the less critical heaters. The precision high impedance wheatstone bridges have been modified (using low thermal soldering techniques) to permit their use as differential bridges. Wagner-earth resistors which were provided have been connected to a guard terminal to provide a driven guard facility for shielded two core, low thermal cables.

A chopper modulated microvoltmeter null-detector of United States manufacture showed low frequency (0.5Hz) oscillations under certain conditions. This was found to be due to beating between harmonics of the chopper oscillator and the 50 Hz mains power frequencies. Calculations indicated an optimum oscillator frequency of 86 Hz for use with 50 Hz power. The 5th harmonic of the mains frequency (250 Hz) and the 3rd harmonic of the oscillator frequency (258 Hz) could then give rise to 8 Hz beats. The 7th harmonic of the mains (350 Hz) and the 4th harmonic of the oscillator frequency (344 Hz) could give rise to 6 Hz beats. The manufacturer recommends 84 Hz for the oscillator frequency, using 60 Hz power. The third harmonic of this oscillator frequency (252 Hz) could give rise to 2 Hz beats with the 5th harmonic of 50 Hz mains power. The oscillations were removed by setting the oscillator frequency to 86 Hz.

5.4 Other Investigations in Progress

Long exposures to a cobalt-60 source have been given to typical thermistors and early absorber heater discs, to determine whether prolonged irradiation may have an adverse effect. Further exposures of this type are under way using an improved jig, and no results are as yet available.

The uniformity of heating of the absorber heater may be investigated. A possible method to do this would seem to be the use of infra red sensitive photographic film in contact with the heater. This may reveal hot spots, which may arise owing to the particulate nature of the graphite/epoxy mixture. Thus the measured resistance between the leads may be the result of only a relatively few parallel and branching paths from one wire to the other. Indeed, the contacts with the wires themselves may only occur at a relatively few points, if the graphite is distributed in relatively large discrete lumps.

Some consideration has been given to the avoidance of possible ground loops in the electronic measuring system, by establishing a careful grounding "tree". This includes the use of isolated-ground mains power distribution boards in the equipment racks, with separate chassis grounding of each item in the rack through heavy copper wires to a common earthing strap.

The physical disposition of the various electronic components of the measuring system, and their interconnections have been considered. There will probably be one rack of highly sensitive equipment located inside the exposure room, containing equipment for measuring the absorber temperature change (rack 2, figure 8). This will keep the most critical leads short, and facilitate the maintenance of constant temperatures and electrostatic fields in the vicinity of this equipment.

Two further racks of equipment are planned in the adjacent measurement laboratory. One (rack 1, figure 8) will probably contain the calibration heater controller, voltmeter and timer, the other (rack 3) will carry the remaining, less critical temperature controllers.

6. SUMMARY

This report describes progress in the development of a calorimetric working standard of ionising radiation absorbed dose.

An outline of the background to the project is given, including a brief history of the quantities and units used in this work. The absorbed dose quantity is discussed in detail, and the requirements for accuracy in ionising radiation dosimetry are discussed.

The general features of the microcalorimeter under development are described. The preliminary results of a literature survey are given, followed by a detailed description of the design of the proposed device. The need for various correction factors is indicated.

Anticipated values of experimental parameters are given, including the absorber temperature rise. The expected method of measuring this rise is described. The means by which the measurement will be made absolute are discussed, including the use of standardised measuring equipment, to allow the absorbed dose to be calculated.

The absorber heater is described in detail. Considerations relating to heat transfer between the various parts of the calorimeter are followed by an indication of the sensitivity required in the electronic equipment used to record the temperature rise due to the radiation.

The philosophy adopted for the temperature control of the various parts of the calorimeter is described.

The effect of electrostatic fields on the high impedance circuitry, and modifications that have been found necessary to certain electronic measuring equipment are described.

The present status of construction is indicated. Testing programs that have been completed, are under way or are planned are discussed; results are given where these are available.

Particular difficulties that have been encountered include the welding of very fine metal wires. The solution to this problem is described in detail.

Radiation damage to the thermistors and heaters and the uniformity of heating provided by the absorber heater are to be investigated.

The arrangement of electronic equipment is indicated and a block diagram of the overall system is given.

7. ACKNOWLEDGEMENTS

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TABLES

1. Properties of Commercially Available Resistance Wire
2. Temperature Control and Heat Transfer

Table 1
Properties of Commercially Available Resistance Wire.

Alloy name, composition (percent)	Z (note 1)	d μm (note 2)	k Wm ⁻¹ K ⁻¹ (note 3)	kA nWmm mK ⁻¹ (note 4)	RL ⁻¹ ohm m ⁻¹ (note 5)	ML ⁻¹ mgm ⁻¹ (note 6)
constantan Cu55/Ni45	29	25	22	11	900	4.4
manganin Cu86/Mn12 /Ni2	29	45	22	34	370	13.3
evanohm Ni75/Cr20/ Al2.5/Cu2.5	25	25	14	7	2700	4.0
nichrome Ni80/Cr20	27	25	13	6	2200	4.1
chromel Ni90/Cr10	28	25	15	7	1500	4.2
alumel Ni95/(Al+Mn +Si)5	27	25	30	15	700	4.3
Pt90/Rh10	76	25	38	19	440	9.8
Pt90/Ir10	78	25	31	15	570	10.6
copper	29	25	400	200	33	4.4
copper	29	100	400	3200	2.1	70

- Notes
1. $Z = \frac{\sum_i f_i A_i Z_i}{\sum_i f_i A_i}$
 2. d = diameter of wire
 3. k = thermal conductivity
 4. kA = linear thermal conductivity
 5. RL^{-1} = resistance per unit length
 6. ML^{-1} = mass per unit length
 7. power transfer along wires = $kA \Delta T/L$

- References
- | | |
|--------------------------|-------------------|
| Collocott (1971) | Weast (1979) |
| Gardner and Cooke (1971) | Goodfellow (1979) |
| Kaye and Laby (1973) | |

Table 2
Temperature Control and
Heat Transfer

part	mass g	allowed temperature fluctuation	maximum power input (see note below)
Absorber	2.5	1 μ K	2 nW
Jacket 1	33	10 μ K	200 nW
Jacket 2	110	100 μ K	10 μ W
Mantle	570	1 mK	400 μ W
Vacuum Vessel		10 mK	
Connector Panel		100 mK	

Note: Power = $E/t = mc \Delta T/t$

$c = 730 \text{ J kg}^{-1} \text{ K}^{-1}$

$t = 1000 \text{ s}$

\therefore Power = $0.7 \text{ m } \Delta T$

FIGURES

1. ARL graphite microcalorimeter
2. Microcalorimeter suspension threads
3. Absorber heater
4. Absorber wiring
5. Sensitivity
6. Capacitance discharge welder
7. Welder work holder
8. Microcalorimeter block diagram

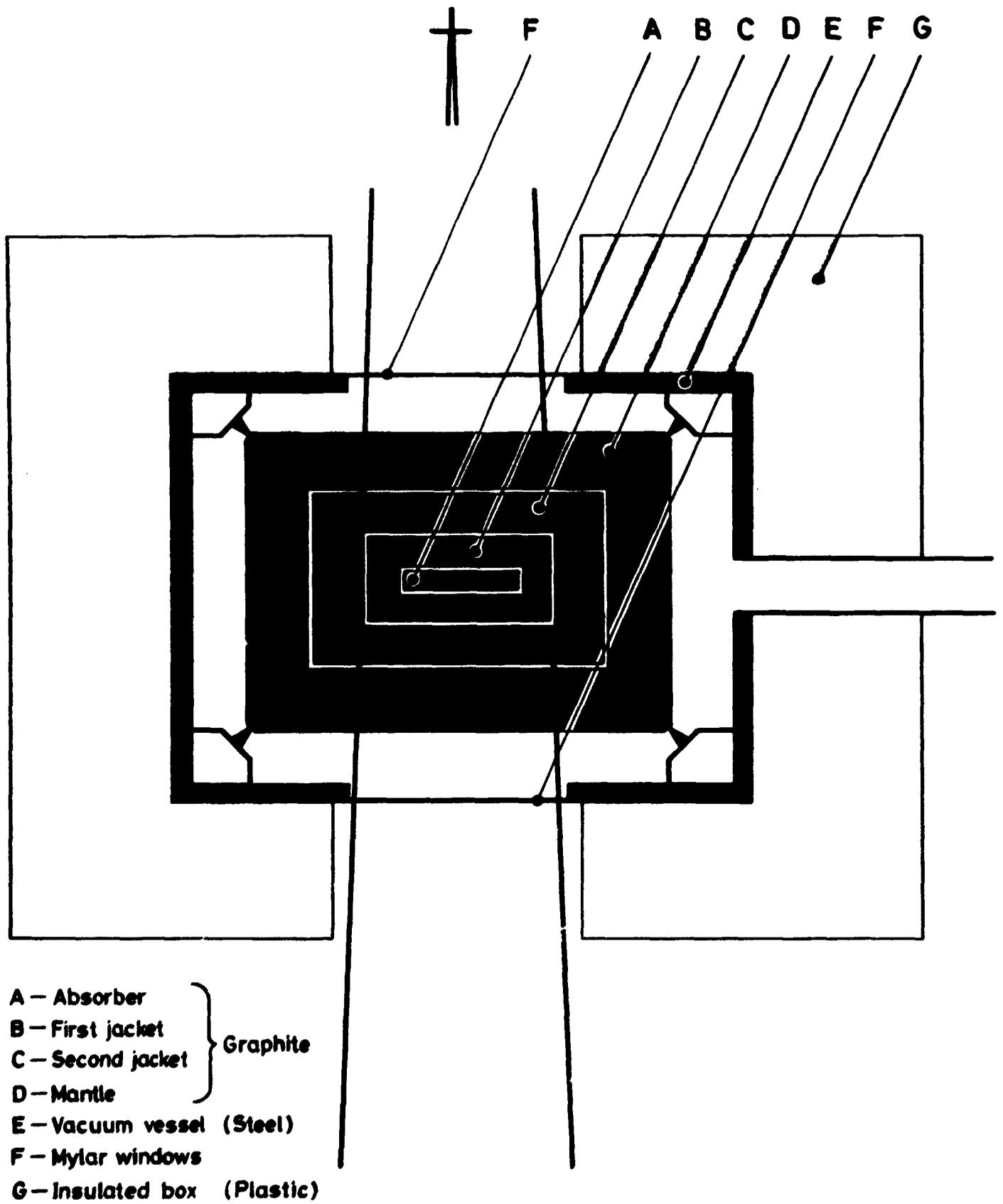
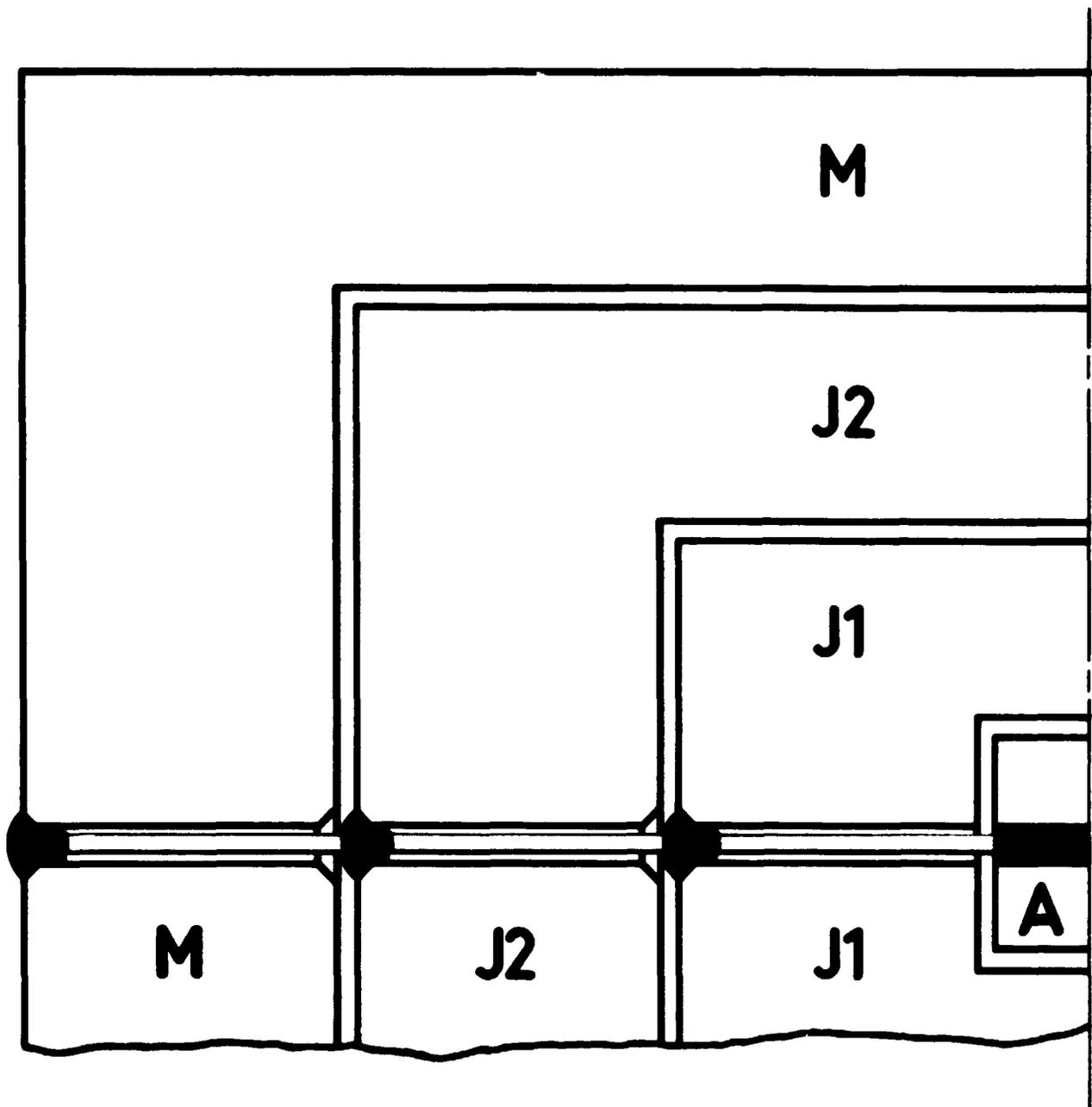


FIG. 1

ARL GRAPHITE MICROCALORIMETER



A — Absorber
J1 — Jacket 1
J2 — Jacket 2
M — Mantle

 **Epoxy resin weld**
 **Suspension thread**

FIG. 2

MICROCALORIMETER SUSPENSION THREADS

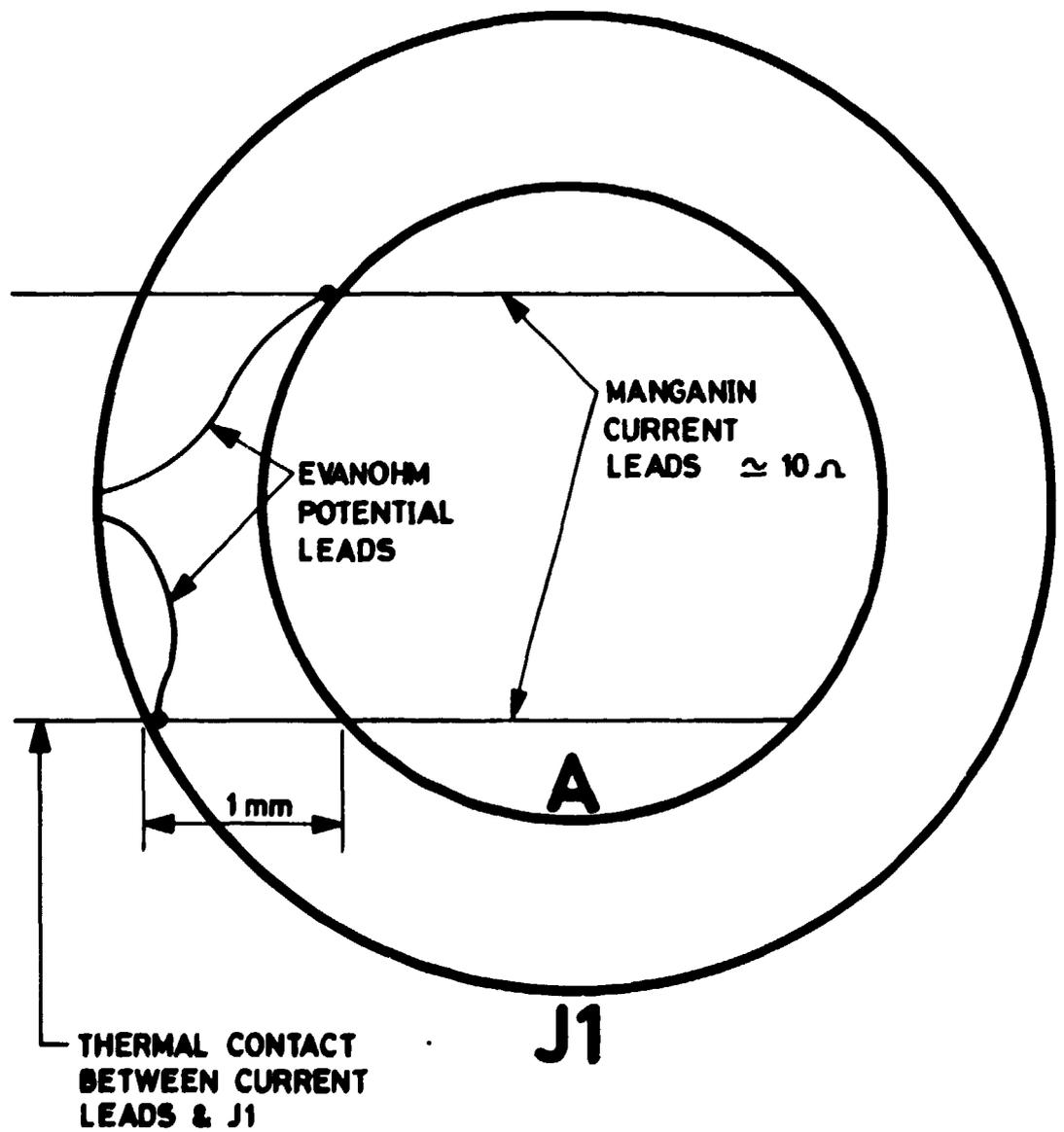
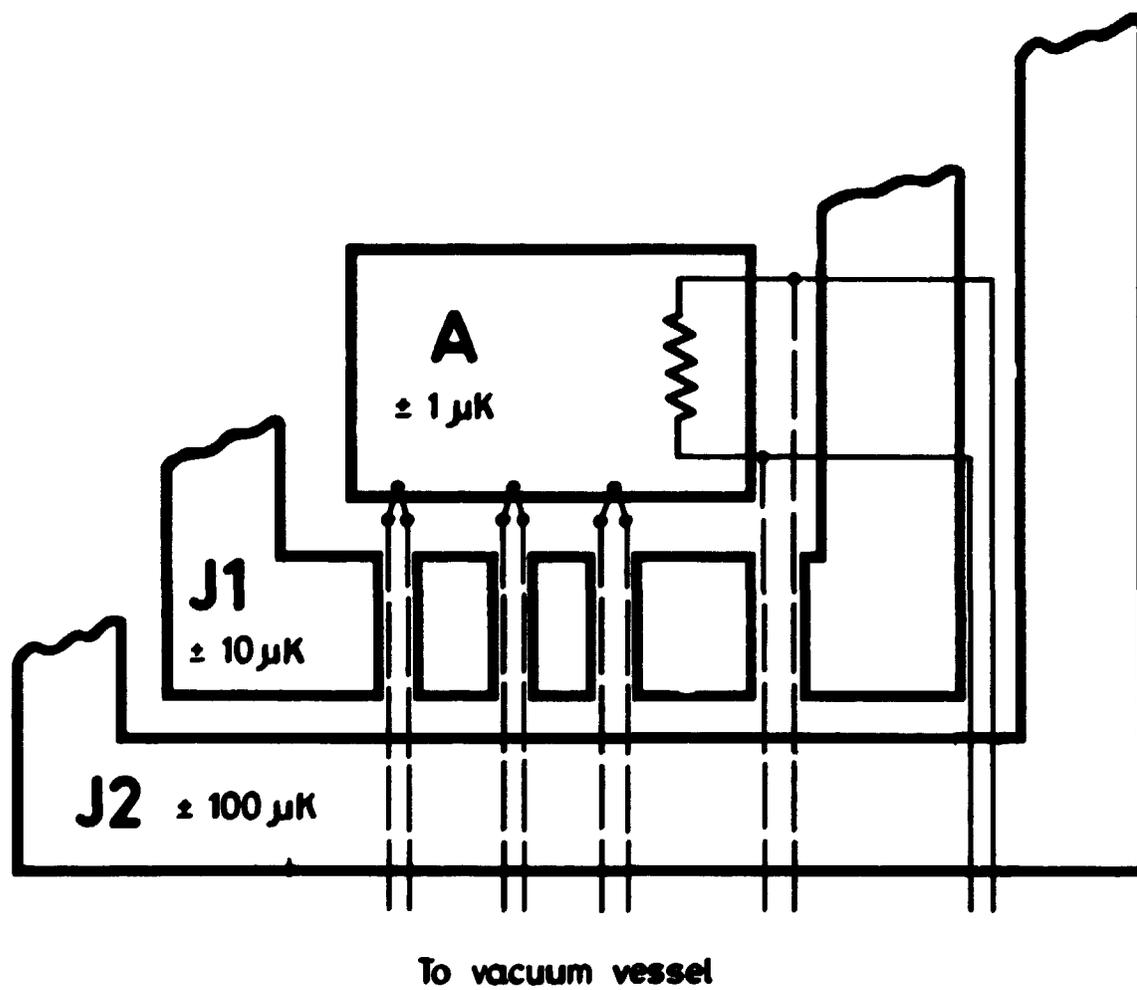


FIG. 3

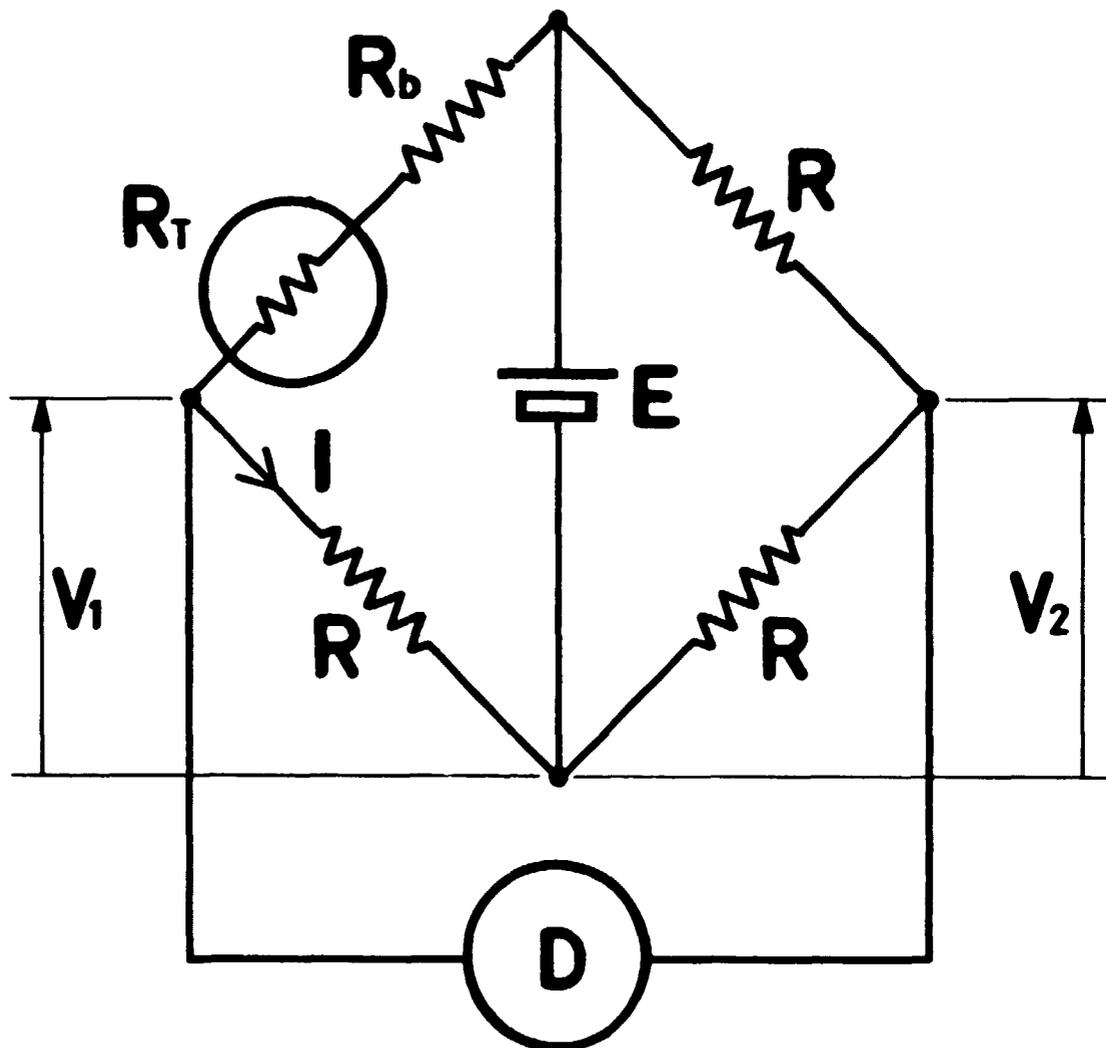
ABSORBER HEATER



— Manganin
 - - Evanohm

FIG. 4

ABSORBER WIRING



At balance ; $R_b + R_T = R$, $V_1 = V_2$, $I = E/2R$

After irradiation ; $R_T \rightarrow R_T + \Delta R$

then , $I \rightarrow E/(R + R_T + R_b + \Delta R) = E/(2R + \Delta R)$

$$\therefore \Delta I = E/(2R + \Delta R) - E/2R$$

$$= E \frac{2R - (2R + \Delta R)}{2R(2R + \Delta R)}$$

$$\approx - \frac{\Delta R}{2R} \cdot \frac{E}{2R}$$

$$\therefore \Delta V_1 = R \Delta I = - \frac{\Delta R}{2R} \cdot \frac{E}{2} = - \frac{\Delta R}{R} \cdot \frac{E}{4}$$

FIG.5

SENSITIVITY

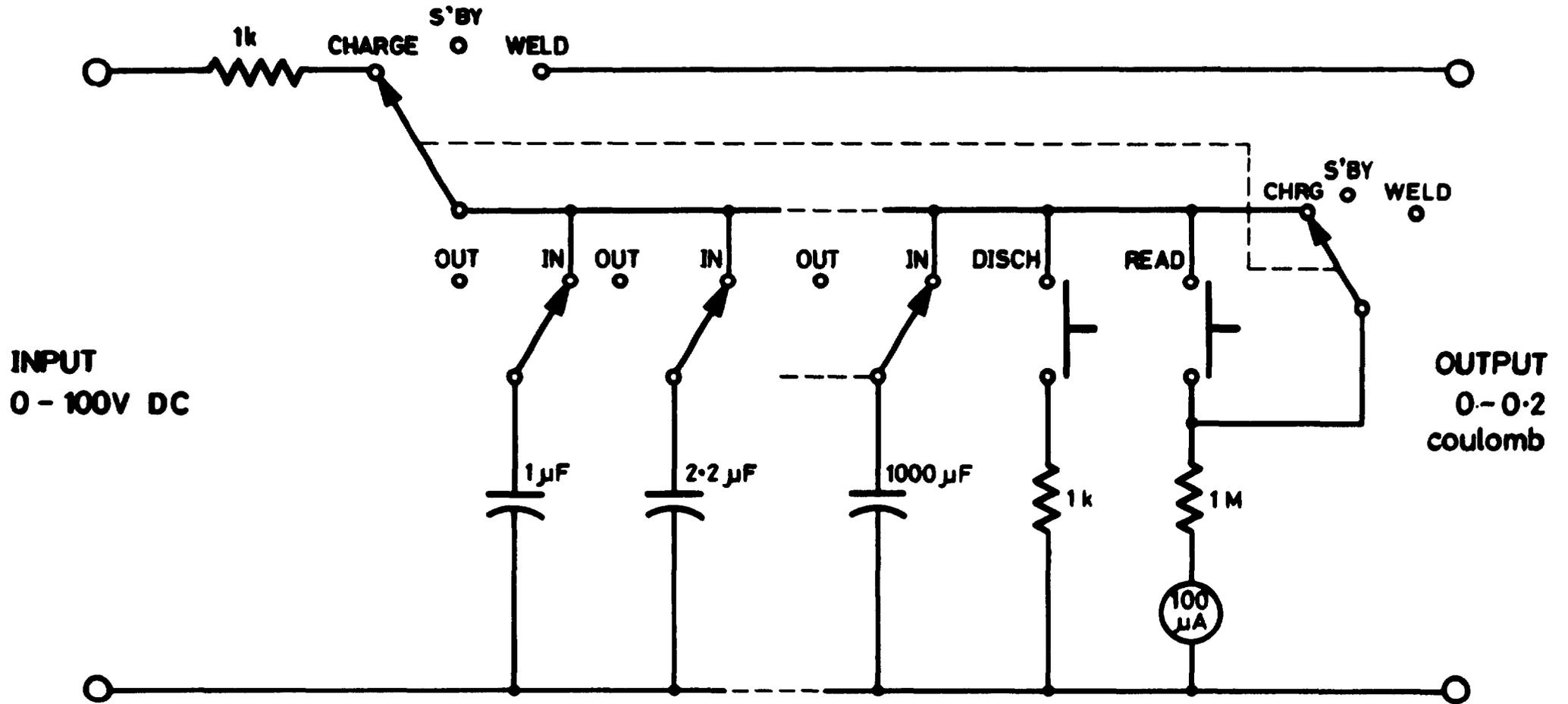


FIG.6

CAPACITANCE DISCHARGE WELDER

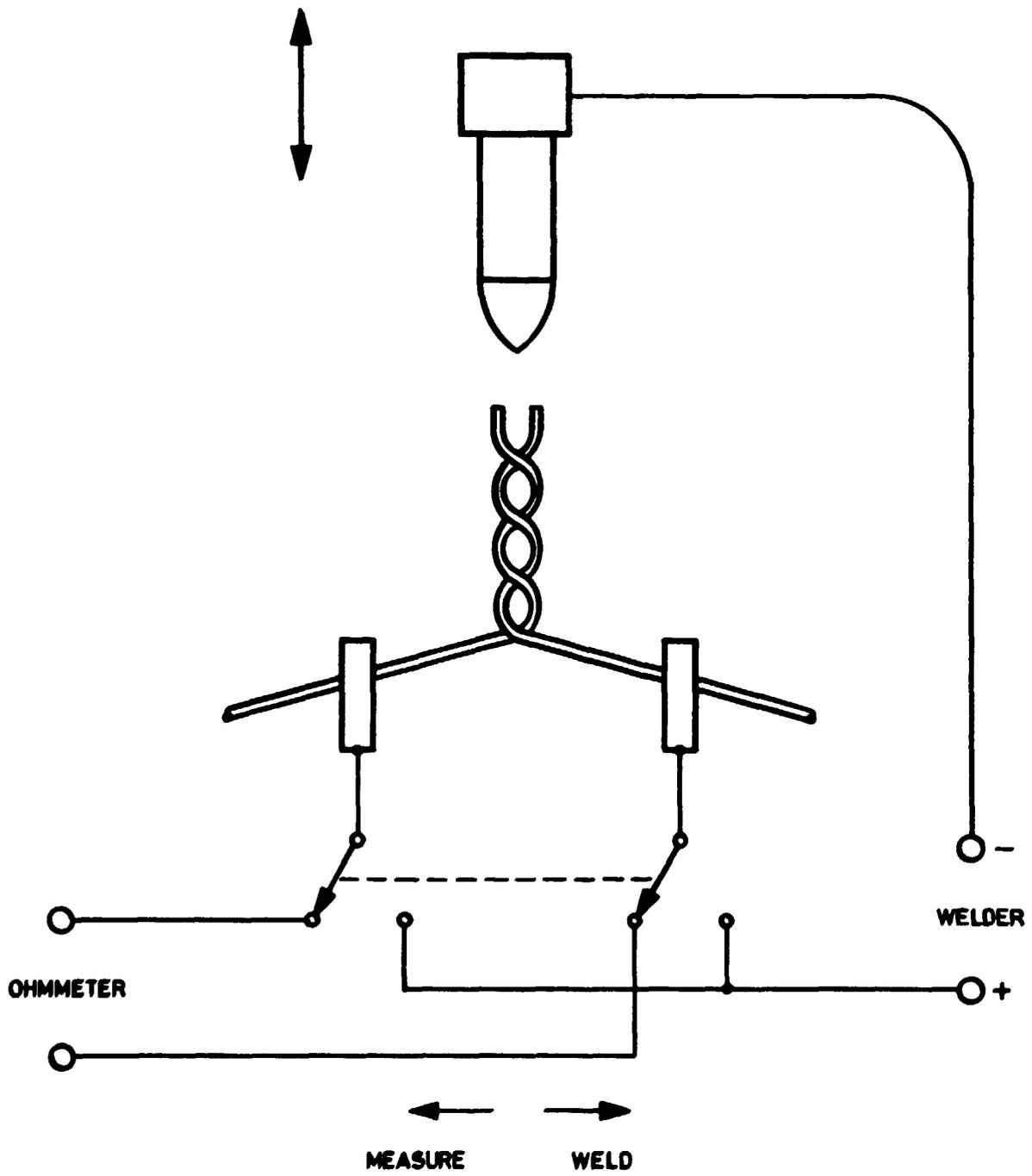


FIG. 7

WELDER WORK HOLDER

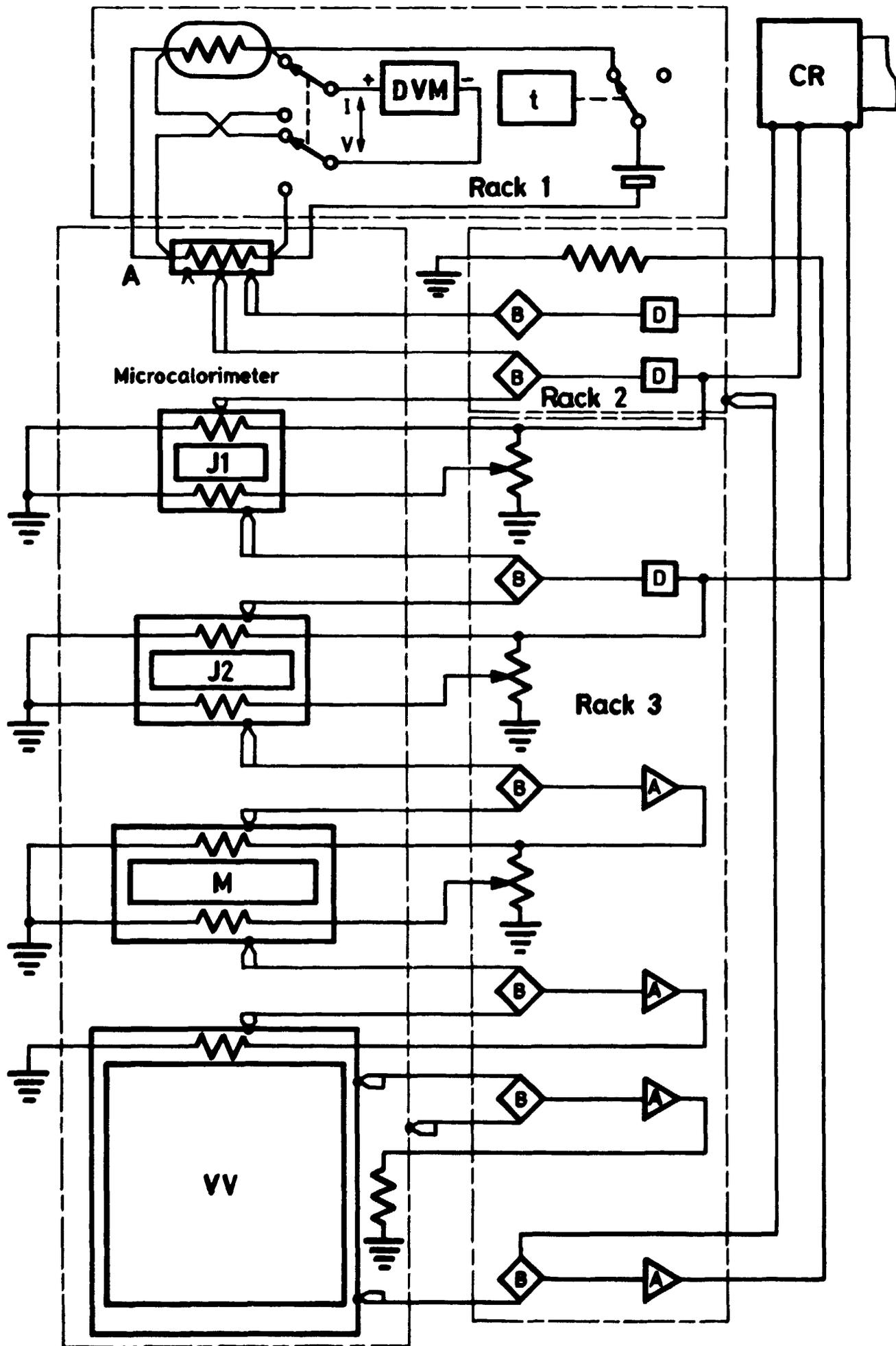


FIG. 8

MICROCALORIMETER BLOCK DIAGRAM