Heavy-water electrolysis unit for generation of deuterium gas provided with automatic switch-off and safety devices

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Abstract

An electrolysis apparatus has been constructed for the safe and trouble-free generation of deuterium gas from heavy water for experimental purposes. It has been found particularly suitable for introducing the gas at a rate of millilitres per minute (S.T.P.) into a vacuum system without the use of an extra pumping unit, because the electrolysis is done under atmospheric pressure and not in a vacuum. The apparatus is equipped with electrical control-relays and needs little attention. It operates within a specified pressure range of the liberated deuterium gas, and is automatically shut off if the pressure exceeds a certain value. There is an almost complete absence of danger of explosion arising from the electrical devices should the generated deuterium gas be accidentally mixed with air.

A SKETCH of the apparatus is given in Fig. 1 and the electrical circuit is shown in Fig. 2. The electrodes for electrolysing heavy water are in the form of platinum foils held in the soda-glass U-tube containing the acidulated heavy water by platinum-wire seals. The deuterium gas generated passes from the moisture absorber* through the Pyrex tube T_1 of 8 mm bore. The tube T_1 is let into a wider tube T_2 of 25 mm bore through a seal in its side wall, and is held concentrically within it. The open end of T_1 rests at about 5 millimetres above the open end of T_2. Both ends are immersed in the mercury contained in the reservoir M. The delivery end E of T_2 is connected to the vacuum system and the rate of entry of the gas is controlled by the needle valve N. At the start, when N is opened to the vacuum system, a mercury column rises in the tube T_2 through the annular space between T_1 and T_2, and assumes the barometric height. Care is taken to ensure that the mercury level within the reservoir M is a few millimetres (3 mm in this case) above the open end of T_1. The deuterium gas generated in T_1 has to build up a pressure equivalent to this few millimetres of mercury above atmospheric pressure in order to pass into the gas container G by displacement of mercury in T_2. As more and more gas is liberated in G, it develops greater pressure, and the mercury column M_1 in T_2 falls. The change in the column height denotes the gas pressure within the container. For operation with this apparatus (the maximum pressure within the deuterium container being 50 cm of mercury) the gas pressure in T_1 was such that the difference in heavy-water levels in the two arms of the U-tube was never more than 9–10 cm. The quantity of liquid in the U-tube must be sufficient to ensure that, under this extreme condition, the electrodes are well immersed in the liquid. To prevent excessive jerky movement of the mercury column in T_2 when the gas escapes through it to the container, glass beads are embedded at places inside its wall.

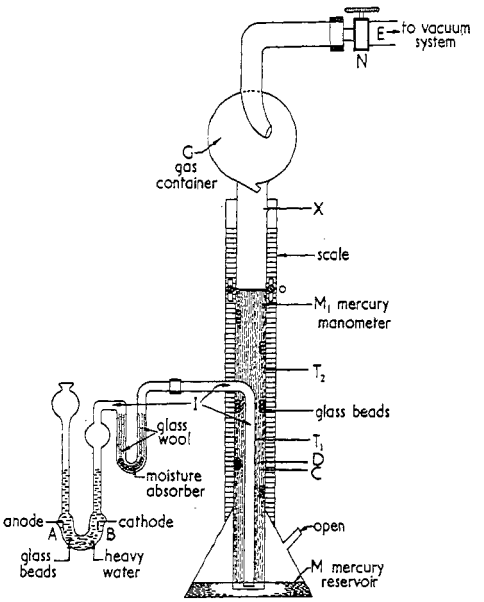

Fig. 1. Heavy-water electrolysis unit

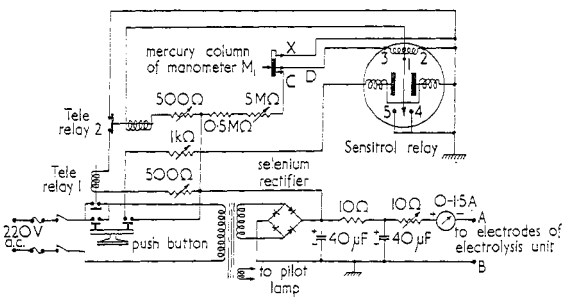

Fig. 2. Automatic control circuit

The tube T_2 is provided with three tungsten wires sealed to it at C, D and X. The mercury column in T_2 serves as a manometer, and also operates the automatic devices by switching actions between C and D and D and X. The electrical circuitry comprises a "Sensitrol" relay (Weston Mod. 705), telephone relays and a 28 V selenium rectifier which supplies power for the electrolysis as well as for the

* In this work, complete freedom from the inevitable trace of oxygen was not required. If it is, a catalytic re-combination agent should be introduced before the moisture absorber.

relays. Electrolysis can be started by pressing the push-button switch (Fig. 2) if the mercury column is above *D*. The push-button is a double-pole switch. One pole serves to complete the primary circuit of the transformer, and the other releases the magnetic needle-clutch of the Sensitrol relay ready for operation. Subsequently, the primary circuit is completed through the contacts of the Tele-relay 1 for all reading positions of the Sensitrol relay needle. The contacts of the Tele-relay 2 are normally closed for operation. When the Sensitrol relay needle is at the end positions, the coil of the Tele-relay 2 is energized and its contacts break, thereby breaking the primary circuit. The circuit will then remain open until and unless the push-button is again pressed for operation.

When the gas pressure inside the container exceeds 50 cm of mercury, the contact between *C* and *D* through the mercury column is broken and the entire electric power supply to the electrolysis unit and the control circuit is disconnected. For operation to restart, the gas pressure has to be reduced to below 50 cm of mercury, and the push-button pressed. Sometimes it may happen, if the gas generation is very rapid, that the upward jerky movement of the mercury column might damage the glass apparatus. To prevent this, as the mercury column makes contact with the tungsten wire at *X* there will be a switch arrangement to short circuit the coil of the Sensitrol relay, and the input power will be cut off as before.

The mercury-column switch carries only currents of the order of a few microamperes flowing through the Sensitrol relay-coil. There is, therefore, little chance of this switching causing an explosion if there is an accidental mixture of air with the generated deuterium gas.

There is one disadvantage with this type of apparatus. When operation is started, some air is trapped in the portion *I* (Fig. 1). This gets diluted gradually with the generated deuterium gas and escapes through the mercury column. After a run of about an hour or so, the portion becomes free from air for all practical purposes and the deuterium gas should only then be collected and stored. In all subsequent runs, however, no question of contamination with air arises, and pure deuterium gas is obtained because *I* always remains filled with the liberated deuterium gas.

There is another way of shutting down the electrolysis unit when the gas pressure in the container exceeds the desired value. This is done by adjusting the amount of the heavy water in the electrolysis U-tube in such a way that, as soon as the gas exceeds that pressure, the electrolyte level in an arm of the tube goes below the electrode housed in it. However, the method does not ensure a dependable and consistent arrangement without external controls as in the previous case. Nevertheless, it does provide an extra precaution in case the control-unit fails. In this apparatus, the amount of heavy water is such that, if the gas pressure exceeds 55 cm of mercury, the electrolyte level goes below one of the electrodes.

In the event of an accident (for example, if the gas-container or the manometer cracks), the mercury goes back to the reservoir and no spilling of the heavy water out of the U-tube can occur.

The apparatus has been supplying deuterium gas to the ion-source of the Calcutta 37 in. Cyclotron for a couple of years and requires little attention for its operation. Normally it runs with an electrolysis current of 1 ampere.

Acknowledgements

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Temperature-controlled permanent magnet for high-resolution nuclear magnetic resonance

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Abstract

The design and construction of a large temperature-controlled permanent magnet is described and the considerations given towards obtaining a high degree of field uniformity. Using current shims and a spinning sample, a resolving power and stability of a few parts in 10^8 *may be obtained at any time and, with care,* 1 *part in* 10^8 *can be achieved.*

Introduction

SEVERAL accounts have been given of magnets for high-resolution nuclear magnetic resonance in which the time stability of the magnetic field is achieved electronically. This paper described a relatively simple high-resolution permanent magnet which provides a good performance without electronic stabilization.

Magnet design

The basic design of the magnet is governed by three main considerations, namely, high field strength, stability and field uniformity.

The signal-to-noise ratio of a nuclear resonance increases as $H^{7/4}$, provided the line width remains constant. If the magnet inhomogeneity exceeds the line width, and is assumed to be proportional to H, then the maximum absorption intensity increases only as $H^{3/4}$. A high field is desirable in order to increase the chemical shift with respect to spin-spin coupling constants. The maximum field is also governed by the gap size. The gap must be large enough to allow a reasonable working space, and to minimize the effects of inhomogeneities in the pole-piece material. A gap of $1\frac{1}{2}$ in. was chosen. Optically flat pole pieces are used of sufficient diameter to give a satisfactory region of high uniformity.