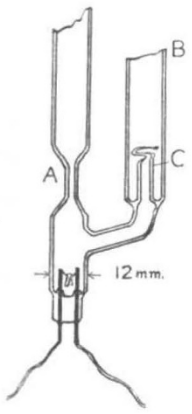
Microsynthesis of C^{14}-Labelled Ethylmagnesium Iodide

THE synthesis of C^{14}-labelled ethyl iodide on the 10–15 micromole scale has been described[1], the scale being chosen so that high specific activity $C^{14}O_2$ could be used undiluted as starting material. The product would then contain about 6 microcuries of carbon-14 per micromole. On about the same scale, radioethyl iodide can without dilution be converted to radioethylmagnesium iodide by reaction in ethereal solution with a magnesium mirror.



The reaction is carried out in a small vessel as shown in the accompanying figure. This reaction vessel is attached to the high-vacuum apparatus; it has a platinum filament on which is hung a small piece of magnesium ribbon. The vessel is dried thoroughly by baking out at 300° C. under vacuum for a total of about thirty hours, and a mirror is then thrown up on the walls of the tube by heating the filament electrically. About 150 standard microlitres of dry radioethyl iodide vapour are condensed in, followed by 10 standard millilitres of very carefully dried diethyl ether vapour, after which the vessel is removed from the apparatus by sealing off at *A*.

The best yield of the Grignard reagent which has been found is about 55 per cent, and this is obtained by incubating the contents of the reaction vessel at 37° C. for two hours: shorter or longer periods of incubation give lower yields for reasons not ascertained. Yield is determined by measuring the ethane formed on decomposition with water. This is done immediately after incubation by resealing the reaction vessel to the apparatus at *B*, breaking the seal *C*, distilling off ether and unreacted ethyl iodide and then condensing water vapour on to the dry residue. The radioethane is purified by a standard technique and, after measurement in a McLeod gauge, is counted in a Brown and Miller type β-counter without first burning to carbon dioxide, as the amount is small and there are no losses due to adsorption on the counter walls.

The specific activity of the ethane is always that expected from the known specific activity of the radioethyl iodide used, indicating that in the conditions of these experiments there is no exchange between the ethyl group of ethyl iodide and those of diethyl ether.

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[1] Arrol and Glascock, *Nature*, **161** 932 (1948).

Poisoning in High-Vacuum Oxide-Cathode Valves

IN the course of our investigations into the causes of deterioration of emission from oxide cathodes. we have noticed a phenomenon of possible significance. The phenomenon has been found to be common to a variety of tubes, including commercial pentodes and valves prepared in our own laboratory. A full investigation will be published in due course; but it is felt that a note at this stage might interest some readers.

It is fairly generally accepted that the life of an oxide-cathode valve is a function of the cathode loading, that is, the current density drawn through the cathode. An inference frequently drawn is that the magnitude of the current traversing the cathode is a direct factor in decreasing the emission of the cathode during life by electrolytic removal of active barium or some other mechanism. Our own conclusion is somewhat different, and follows the line that the magnitude of the current is of little importance within fairly wide limits; but that the ionizing action of the current *after* leaving the cathode surface is the prime factor leading to cathode deterioration. An illustration may help to make clear our meaning.

A commercial high-slope oxide-cathode pentode valve type with a known life of 500–1,000 hr. was selected for the experiment. Two batches of eight valves each were taken at random and placed on life-test in the following manner. Tubes of batch *A* were arranged as diodes with control grids acting as collectors and a positive voltage (approximately 2 volts) applied between grid and cathode to give a cathode current of approximately 12 mA. Tubes of batch *B* were arranged as triodes with anodes strapped to screen and suppressor grids and primed with 200 volts. Control grid voltages of the triodes were adjusted to allow each tube to pass 6–8 mA. Cathode temperatures of both batches were maintained at a nominal 1,000° K. Life-test of the valves showed that the diode-connected batch showed little deterioration of diode current over 6,000 hr., whereas the triode-connected batch failed in 500–1,000 hr. A typical example is shown in Fig. 1.

The phenomenon can be shown in effective fashion with a diode and triode assembled in the same envelope using opposite sides of a common cathode. Results from such a tube proportioned specially for

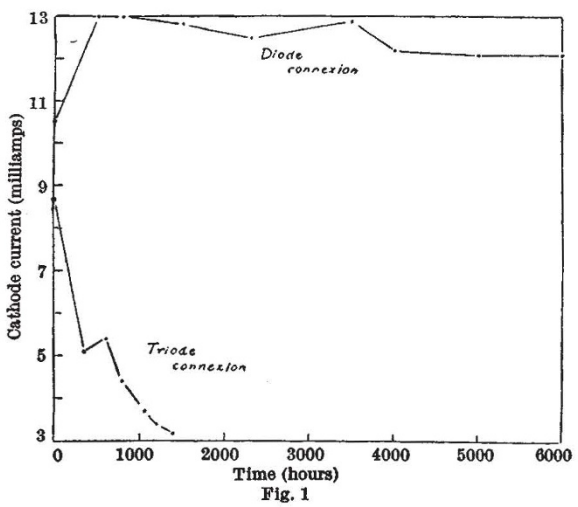

Fig. 1